August 1955 Pages 433-488

The Journal
of the
SOCIETY OF DYERS AND COLOURISTS

Volume 71 Number 8

CONTENTS

PROCEEDINGS OF THE SOCIETY—
Some Observations on the Dyeing of Wool at High Temperatures (*G. A. Coutie, D. R. Lemin, and H. Sagar*) 433
The Dyeing of Textile Fibres above 100°C. (*J. A. Fowler*) 443
Courplsta—The Dyeing and other Properties of Cellulose Triacetate Yarn and Staple (*J. Boulton*) 451
ERRATA 464
NOTES 465
NEW BOOKS AND PUBLICATIONS 466
ABSTRACTS 469

THE SOCIETY OF DYERS AND COLOURISTS
19 PICCADILLY · BRADFORD 1 · YORKSHIRE

Excellent for ALL TEXTILE Processes

GARBRITOL SE
for
Scouring
Dyeing
Finishing

FIRST IN QUALITY

Brilliant Avirols
for a
Quality Handle
on
Rayons

THE GARDINOL CHEMICAL CO LTD Milnsbridge Huddersfield

Famous Links

Staveley Chemicals are the "Links" between raw materials and finished products in industry

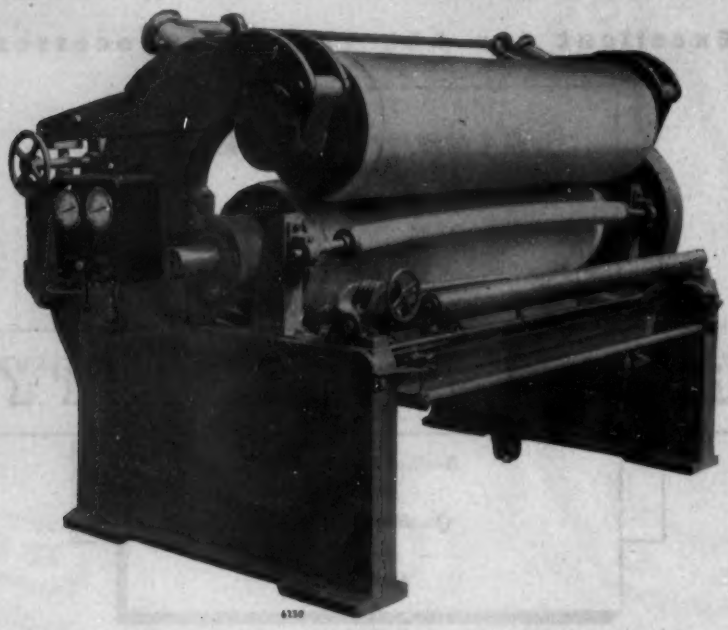
THE OLD COURSE AT ST. ANDREW'S
A VIEW OF THE 18th HOLE

SALT — CAUSTIC SODA — TEXTILES
SULPHUR — OLEUM — SYNDETS
STAVELEY

BASIC CHEMICALS FOR INDUSTRY

THE STAVELEY IRON & CHEMICAL CO LTD NEAR CHESTERFIELD

A



two-bowl pneumatic off-set

Padding Mangle

- Bowls of large diameter for even padding, minimising the wear on the rubber surfaces
- A skewing arrangement for the top bowl to ensure level expression at all loads
- Strong and easily removable roller bearing blocks
- A stainless-steel heated trough of small capacity with an automatic dye-level control. The trough is designed for even feeding of the dye liquor
- A quick-action "On-Off" device for pressures up to six tons
- Independent pressure adjustment for each side of the machine

MATHER & PLATT LIMITED

PARK WORKS · MANCHESTER 10

509

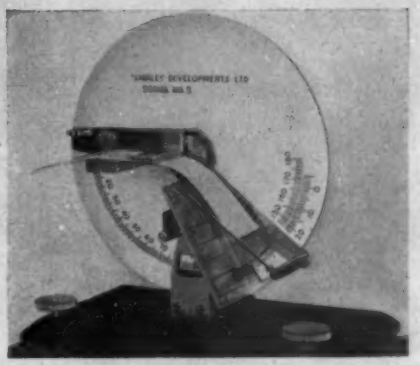
Cut down trial and error — get the facts with a

'SHIRLEY' COMBINED CREASING & STIFFNESS TESTER

. . . by using the widely accepted procedure devised by the Shirley Institute for measuring, specifying and comparing the stiffness, creasability, or crease-resistance of fabrics. The effects of varying quantities and types of crease-resistant and other additives on these properties can be compared and specified.

Important — All "SHIRLEY" instruments are carefully checked at the Institute for performance and workmanship before delivery.

Early delivery — Ask for our folder SDL/3



SHIRLEY DEVELOPMENTS LIMITED

40 KING STREET WEST MANCHESTER 3 *Telephone* **DEAnsgate 5926** *and* **8182**

The word "SHIRLEY" is a trade mark and is the property of The British Cotton Industry Research Association

Laporte Progress

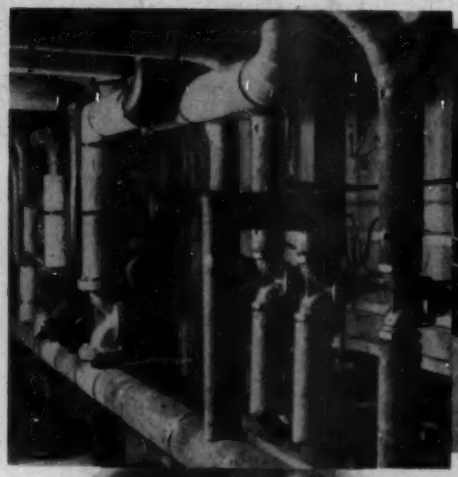
1898

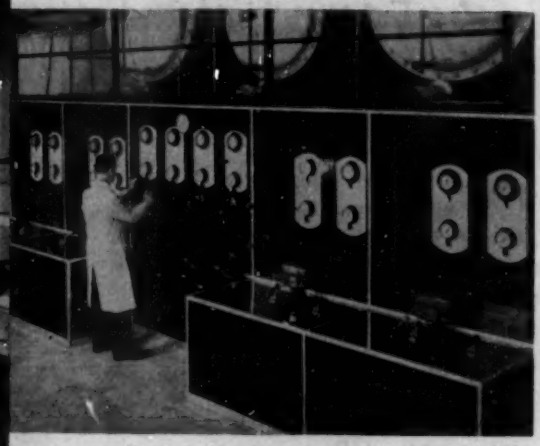
1898 had its traffic problems too— with steam buses and the new-fangled motorcars jostling horse-drawn buses and cabs into confusion. Industry was still striving ahead. Laporte now had a branch works at Luton to supply the straw plait industry with hydrogen peroxide, a further development of this product for industry. The House of Laporte has held the lead since 1888 and application of hydrogen peroxide now varies from bleaching textiles to a compact source of concentrated energy for driving torpedoes, submarines, rockets and for assisted take-off for aircraft.

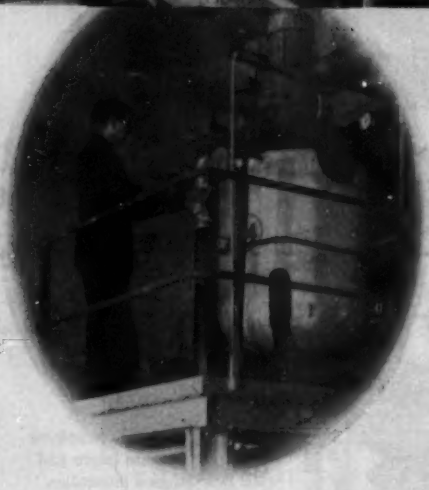
HYDROGEN PEROXIDE

by **LAPORTE**

Laporte Chemicals Ltd, Luton *Telephone: Luton 4390* *Telegrams: Laporte, Luton*







Some of the 165

SAUNDERS DIAPHRAGM VALVES

installed in the FINA Lubricating Oil Blending Plant, Millwall

Appreciated here and throughout industry for their sure closure by flexible diaphragm; for their isolation of mechanism from the fluids under control; for the exactness with which they can be adapted to difficult and dangerous fluids and for their simplicity of maintenance and consequent long life

1 Behind the main control panel, screwed and flanged valves handling base oil in final stages of processing. Valves equipped with extended spindles for remote control

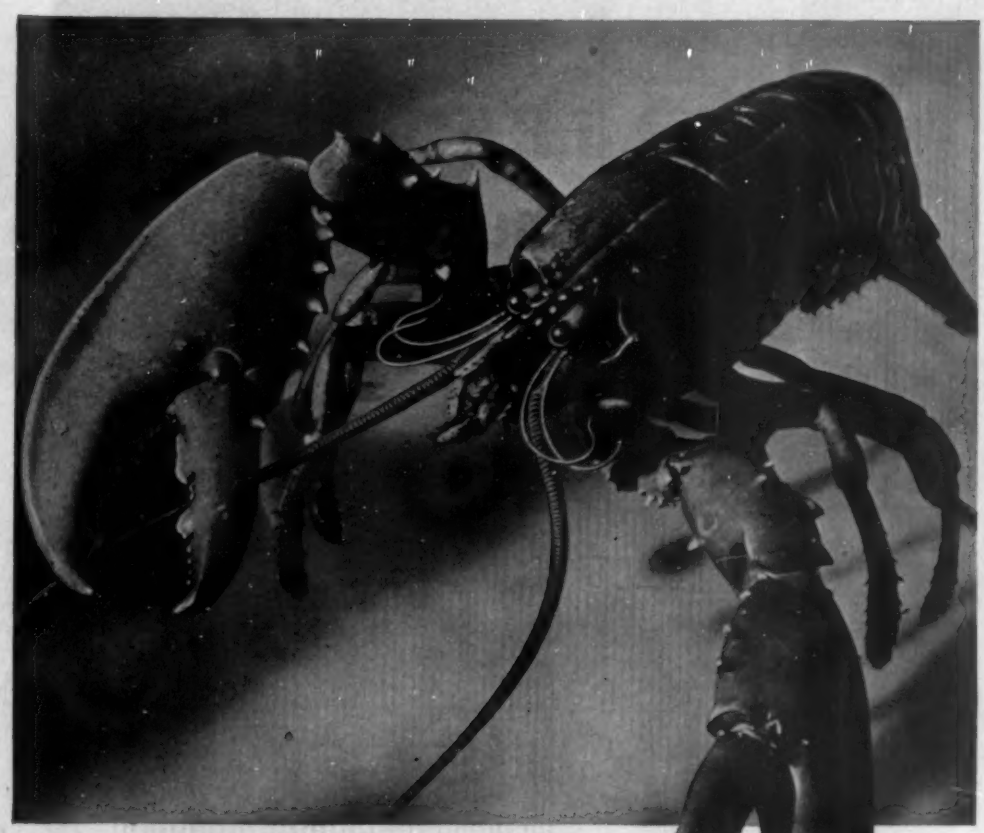
2 The control panel showing 20 of the 50 Saunders Valves in this section of the plant

3 Saunders Diaphragm Valve on one of the many oil blending tanks in the factory

SAUNDERS VALVE COMPANY LIMITED

Diaphragm Valve Division

CWMBRAN NEWPORT MONMOUTHSHIRE



*A Lobster's not the best Chelating Agent **

******The phrase Chelating Agent is derived from the word chela, meaning the prehensile claw of a crab, lobster or scorpion. (But you will find "Metaquest" very much better suited to your needs.)*

The three types of " Metaquest " complexing agents are the most efficient general-purpose sequestering agents yet discovered. They are all compounds of ethylene diamine tetra-acetic acid. If you are processing textiles, cosmetics, pharmaceuticals, or any other product in which soluble metal impurities are a problem, write to Whiffens about " Metaquest " sequestering agents.

There are three types of "Metaquest"

"METAQUEST" A
ethylene diamine
tetra-acetic acid (E.D.T.A.)

"METAQUEST" B
di-sodium salt of E.D.T.A.

"METAQUEST" C
50% W/V solution tetra-sodium
salt of E.D.T.A.

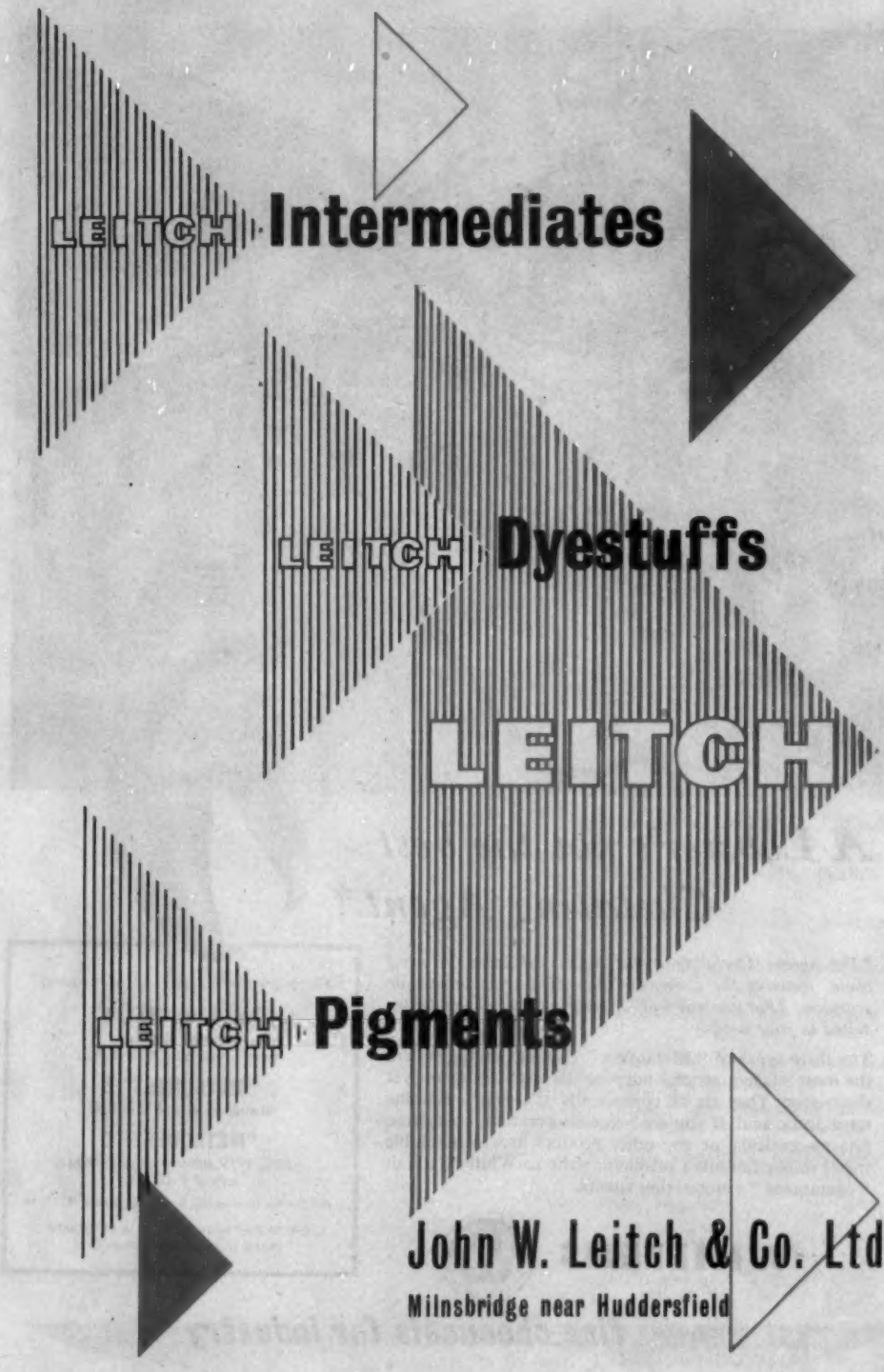
For further information get in touch with Whiffens

THE WORD "METAQUEST" IS A REGISTERED TRADE MARK OF GENATOSAN LTD.

WHIFFENS

fine chemicals for industry

WHIFFEN & SONS LTD., NORTH WEST HOUSE, MARYLEBONE ROAD, LONDON, N.W.1. TEL: PADDINGTON 1041/9. GRAMS: WHIFFEN, NORWEST, LONDON


LEITCH Intermediates
LEITCH Dyestuffs
LEITCH
LEITCH Pigments
John W. Leitch & Co. Ltd
Milnsbridge near Huddersfield

FOR DYEING AND 'WETTING-OUT' OF ALL FABRICS

NEWTRELL P.E.N

We supply large quantities of NEWTRELL P.E.N to COURTAULDS LTD

An extremely efficient scouring, penetrating and levelling agent, Newtrell P.E.N is highly concentrated but quite safe even to the most delicate fabrics and effective in hot or cold solutions. See for yourself—ask us for literature and samples

Quotations given c.i.f. any world port

NEWTRELL

Other NEWTRELL products.

NEWTRELL 'C'—For boiling and dyeing of cotton, linen, yarn, and piece goods.

NEWTRELL 'W'—For scouring and dyeing woollens, worsted, knitwear, etc.

NEWTRELL 'MER'—Valuable aid to the penetration of caustic liquor in mercerising.

NEWTRELL 'SM'—Ideal for the removal of spots of grease, tar, paint, etc, from finished goods.

NEWELL (CHEMICALS) LTD NEWTRELL WORKS HAWICK SCOTLAND

Telephone Hawick 2527 Telegrams WAVERLEY HAWICK

LITMUS

PURE GRAN

Litmus Books and a full range of Indicator Papers

INQUIRIES INVITED

JOHNSONS OF HENDON LTD LONDON NW4 Established 1743

A warm handle

Stearate Emulsion A gives a warm handle to "cold" fibres such as Nylon, a full soft handle to woollens, and a soft finish to cottons and rayons.

PHYSICAL PROPERTIES

Base	CYCLOHEXANOL STEARATE
Solid Content	25%
Appearance	THICK CREAM
pH.	4.0 APPROX.
pH. Stability	3.5—5.0
S.G. (20°C.)	0.883

CHARACTERISTICS

Stearate Emulsion A is an aqueous emulsion of Cyclohexanol Stearate that can be applied to all textiles. It is applied after dyeing, during the rinse, and is substantive to Nylon and wool. Samples and all the technical details you may require will be sent to you on request.

STEARATE EMULSION A

Another technical chemical by

HOWARDS OF ILFORD

HOWARDS OF ILFORD LTD · ILFORD · ESSEX
TELEPHONE: ILFORD 3333

A TBW 181

Textile Assistants
sovatex
sufatol
Sovatex ensures the removal of mineral oil and difficult to clear soiling matter in every fabric cleansing process.
Sufatol sulphated fatty alcohols, having a wide adaptability to assist in the better processing of textiles.
STANDARD CHEMICAL COMPANY
CHEADLE CHESHIRE

COLNE VALE
DYE & CHEMICAL CO LTD
MANUFACTURERS OF

BISMARCK BROWN R and Y Conc. and Base	NIGROSINE (Spirit Soluble)	SOLUBLE BLUE R S Conc.
PURE CHRYSOIDINE Y D and R D and Base	NIGROSINE BASE	SOLUBLE BLUE A Conc.
INDULINE (Water Soluble)	BASIC MAGENTA Pdr. and Crys.	INK BLUE N and B N
INDULINE (Spirit Soluble)	ROSANILINE BASE	SOLUBLE BLUE Special Conc
INDULINE BASE	ACID MAGENTA Conc.	SPIRIT BLUE
NIGROSINE (Water Soluble)	HELVETIA BLUE Conc.	INDIGO CARMINE C V Ex.
	PURE SOL. BLUE 3 B Conc.	LAUNDRY BLUES
		METACHROME MORDANT

Also full range of ACID, BASIC, DIRECT and CHROME COLOURS
Samples and Prices will be forwarded on application

MILNSBRIDGE HUDDERSFIELD
Telegrams ANILINE HUDDERSFIELD Telephone Milnsbridge 3

WANTED
COPIES OF BOUND VOLUME
OF
SYMPOSIUM ON FIBROUS PROTEINS
These are urgently required for repurchase by
THE SOCIETY OF DYERS AND COLOURISTS

The Journal of the Society of Dyers and Colourists

(Subscription rates for non-members £4 per annum, post free)

(Abstracts section only printed on one side of paper—£1 10s 0d per annum)

NOTICE TO MEMBERS AND SUBSCRIBERS

Readers requiring *general information* regarding the Official Notices, List of Officers of the Society, etc. should consult pages 1–6 of the January 1955 and pages 369–373 of the July 1955 issues of the *Journal*, or write to *The General Secretary*, The Society of Dyers and Colourists, Dean House, 19 Piccadilly, Bradford 1, Yorkshire (*Telephone* Bradford 25138–9). *Editorial Communications* should be addressed to *The Editor*, at the same address.

Forthcoming Papers

The following papers have been accepted by the Publications Committee, and will appear in future issues of the *Journal* —

LECTURES

The Chemistry of Leuco Vat Dye Esters — *S. Ainsworth and A. Johnson*

Wear Properties of Resin-finished Spun Rayon Fabric — A New Method of Laboratory Assessment — *L. F. H. Breens and T. H. Morton*

The Dyeing of Terylene Polyester Fibre with Disperse Dyes above 100°c. — *A. S. Fern*

The Guilds of the City of London with special reference to the Dyers' Company — *P. E. Jones*

Cellulose Acetate and Water Repellency — *R. J. Mann*

COMMUNICATIONS

The Dyeing of Acetate Rayon with Disperse Dyes. IV — Adsorption Isotherms — *C. L. Bird and F. Manchester*

The Polarography of Azo Dyes — *J. de O. Cabral and H. A. Turner*

The Bleaching of Jute with Hydrogen Peroxide — *H. Chatterjee and K. B. Pal*

The Adsorption of Some Organic Vapours by Wool and Nylon — *H. R. Chipalkatti, V. B. Chipalkatti, and C. H. Giles*

The Iodination of Wool — *H. R. Richards and J. B. Speakman*

The Irgalan Dyes — Neutral-drawing Metal-complex Dyes — *G. Schetty*

CHAS. FORTH & SON LIMITED

CHEMICALS for Dyers & Bleachers
ACIDS ALKALIES SOAPS

DYESTUFFS for all purposes
DYEWOOD EXTRACTS HEMATINES

Telephones 75147—75148
Code A B C 5th Edition

NEW BASFORD NOTTINGHAM

Telegrams DELTA-NOTTINGHAM

For cotton and rayon blends...

TEEPOL

detergent for the textile industry

Wherever the wet processing of cotton and rayon mixtures is involved, the exceptional wetting, penetration and dispersing powers of TEEPOL may be used with maximum benefit. TEEPOL, a Shell product, is ideally formulated to the special and diverse needs for detergency. Full information is freely available and the Shell Technical Service is always pleased to advise on specific problems.

SHELL (DISTRIBUTORS) **Shell Chemicals Limited,** Norman House, 105-109 Strand, London, W.C.2. *Tel: Temple Bar 4455*

Divisional Offices: Walter House, Bedford St., London, W.C.2. *Tel: Temple Bar 4455.* 43 Deansgate, Manchester. *Tel: Deansgate 6451.* Clarence Chambers, 39 Corporation St., Birmingham 2. *Tel: Midland 6934.* 28 St. Enoch Square, Glasgow, C.1. *Tel: Glasgow Central 9561.* 83 Middle Abbey St., Dublin. *Tel: Dublin 45775.* 35-37 Boyne Square, Belfast. *Tel: Belfast 20081*

C.R1

"TEEPOL" is a Registered Trade Mark

FORTHCOMING MEETINGS OF THE SOCIETY

Tuesday, 27th September 1955

SCOTTISH SECTION. *High Temperature Dyeing of Cellulosic and Synthetic Fibres.* Dr. A. Schmitz (Bayer Co.). St. Enoch's Hotel, **Glasgow.** 7 p.m.

Wednesday, 5th October1955

MIDLANDS SECTION. *The Dyeing of Orlon and Orlon Mixtures.* I. M. S. Walls, Esq., B.Sc., Ph.D. (Imperial Chemical Industries Ltd.). Kings Head Hotel, **Loughborough.** 7 p.m.

Friday, 7th October 1955

LONDON SECTION. *The Contribution of Petroleum Chemicals to the Textile Industry.* H. Bratherton, Esq. (Shell Petroleum Co. Ltd.). The Royal Society Rooms, Burlington House, Piccadilly, **London, W.1.** 6 p.m.

BRADFORD JUNIOR BRANCH. *Dyeing Theory in Retrospect.* Dr. J. Simons (I.C.I. Ltd.). Technical College, **Bradford.** 7.15 p.m.

Tuesday, 11th October 1955

SCOTTISH SECTION. *Textile Research— The Problems of Different Fibres.* H. Corteen, Esq., M.Sc., F.T.I., F.Inst.P. St. Enoch's Hotel, **Glasgow.** Joint Meeting with the Textile Institute. 7 p.m.

Wednesday 12th October 1955

NORTHERN IRELAND SECTION. *The Dyeing of the newer Synthetic Fibres.* Dr. I. M. S. Walls (Imperial Chemical Industries Ltd.). Thompson's Restaurant, Donegall Place, **Belfast.** 7.30 p.m.

Friday, 14th October 1955

WEST RIDING SECTION. Supper and Smoker. Victoria Hotel, **Bradford.**

Monday, 24th October 1955

BRADFORD JUNIOR BRANCH. *Fading — Its Nature and Assessment.* H. B. Mann, Esq., M.Sc. (Courtaulds Ltd.). Technical College, **Bradford.** 7.15 p.m.

Tuesday, 25th October 1955

SCOTTISH SECTION. *Kier Boiling.* C. Garrett, Esq., F.T.A. St. Enoch's Hotel, **Glasgow.** 7 p.m.

Wednesday, 26th October 1955

MIDLANDS SECTION. *Textile Bleaching with Peroxide and Peracetic Acid.* L. Chesner, Esq., B.Sc., A.T.I., and G. C. Woodford, Esq. College of Technology, **Leicester.** 7 p.m.

Thursday, 27th October 1955

NORTHERN IRELAND SECTION. *Fire Prevention Methods in the Textile Industry.* Film will be shown. J. R. Coulter, Esq. Thompson's Restaurant, Donegall Place, **Belfast.** Joint meeting with Textile Institute. 7.30 p.m.

WEST RIDING SECTION. *The Dyeing and Finishing of 'Courpleta'.* W. R. Beath, Esq., and J. S. Ward, Esq. (Courtaulds Ltd.). Victoria Hotel, **Bradford.** 7.30 p.m.

Friday, 4th November 1955

LONDON SECTION. *Microscopical Investigations of Fibre Damage.* G. G. Taylor, Esq., and J. C. Brown, Esq. (Clayton Dyestuffs Co. Ltd.). Joint Meeting with the London Centre of the Guild of Dyers and Cleaners. The Royal Society Rooms, Burlington House, Piccadilly, **London, W.1.** 6.30 p.m.

Tuesday, 8th November 1955

BRADFORD JUNIOR BRANCH. *Microscopical Investigations of Fibre Damage.* G. G. Taylor, Esq., and J. C. Brown, Esq. (Clayton Dyestuffs Co. Ltd.). Technical College, **Bradford.** 7.15 p.m.

Wednesday, 9th November 1955

NORTHERN IRELAND SECTION. *Causes of Felting in Rayon Textiles.* H. W. Best-Gordon, Esq. (Courtaulds Ltd.). (Joint Meeting with the Textile Institute), Thompson's Restaurant, Donegall Place, **Belfast.** 7.30 p.m.

Saturday, 12th November 1955

MIDLANDS SECTION. Ladies' Evening— Dinner Dance. Welbeck Hotel, **Nottingham.**

Tuesday, 22nd November 1955

SCOTTISH SECTION. *The Dyeing and Finishing of 'Courpleta'.* W. R. Beath, Esq., and J. S. Ward, Esq. (Courtaulds Ltd.). St. Enoch's Hotel, **Glasgow.** 7 p.m.

Wednesday, 23rd November 1955

MIDLANDS SECTION. *Modern Methods of Finishing Open-width Knitted Fabrics.* Film Evening introduced by J. Dalglish, Esq. Technical College, **Nottingham.** 7 p.m.

BRADFORD JUNIOR BRANCH. *Properties of Terylene/Wool Blends in Yarn and Fabric Form.* B. I. Brooke, Esq. (I.C.I. 'Terylene' Council). Technical College, **Bradford.** 7.15 p.m.

Friday, 2nd December 1955

LONDON SECTION. *The Application and Evaluation of Optical Bleaching Agents.* G. R. Perdue, Esq. (British Launderers Research Association). The Royal Society Rooms, Burlington House, Piccadilly, **London, W.1.** 6 p.m.

Wednesday, 7th December 1955

MIDLANDS SECTION. *The Dyeing and Finishing of 'Courpleta' Knitted Fabrics.* S. N. Bradshaw, Esq. Bell Hotel, **Leicester.** 7 p.m.

Tuesday, 13th December 1955

SCOTTISH SECTION. *Basic Mechanism in Jigger Dyeing of Woven Fabrics.* Dr. J. Ashworth (Courtaulds Ltd.). St. Enoch's Hotel, **Glasgow.** 7 p.m.

Wednesday, 14th December 1955

NORTHERN IRELAND SECTION. *Recent Developments in Bleaching, Dyeing and Finishing Machinery.* K. Laurie, Esq. (John Dalglish & Co. Ltd.). Thompson's Restaurant, Donegall Place, **Belfast.** 7.30 p.m.

Thursday, 15th December 1955

BRADFORD JUNIOR BRANCH. *Experience in the Application of Automatic Regulation to the Dyeing Process.* F. Blezzard, Esq. (Drayton Regulator & Instrument Co. Ltd.). Technical College, **Bradford.** 7.15 p.m.

Wednesday, 11th January 1956

NORTHERN IRELAND SECTION. *The Dyeing of Terylene Polyester Fibres.* H. R. Hadfield, Esq., M.Sc. (Tech.) (Imperial Chemical Industries Ltd.). Thompson's Restaurant, Donegall Place, **Belfast.** 7.30 p.m.

Friday, 13th January 1956

LONDON SECTION. Details later.

BRADFORD JUNIOR BRANCH. Lecture Title to be announced later. Midland Silicones Limited. Technical College, **Bradford.** 7.15 p.m.

Tuesday, 17th January 1956

SCOTTISH SECTION. *What Dyeing does to Wool.* F. F. Elsworth, Esq., Ph.D., A.R.I.C., F.S.D.C. (Wool Industries Research Association). St. Enoch's Hotel, **Glasgow.** 7 p.m.

Wednesday, 18th January 1956

MIDLANDS SECTION. Discussion Group on *Detergency.* Questions invited. G. H. Rostron, Esq., A.R.I.C. (Soap), R. C. Tarring, Esq., B.Sc., A.R.C.S. (Synthetics), and R. S. Hartley, Esq., A.R.I.C. (Research). Kings Head Hotel, **Loughborough.** 7 p.m.

Monday, 30th January 1956

BRADFORD JUNIOR BRANCH. *A New Approach to High Temperature Dyeing.* S. N. Bradshaw, Esq. (Courtaulds Coventry Ltd.). Technical College, **Bradford.** 7.15 p.m.

Thursday, 2nd February 1956

SCOTTISH SECTION. *Scouring and Milling.* B. F. J. Moxon, Esq. (Wool Industries Research Association). The Scottish Woollen Technical College, **Galashiels.** 7 p.m.

Friday, 3rd February 1956

LONDON SECTION. *A New Approach to High Temperature Dyeing.* S. N. Bradshaw, Esq. (Courtaulds Ltd., Coventry). Royal Society Rooms, Burlington House, Piccadilly, **London, W.1.** 6 p.m.

Wednesday, 8th February 1956

NORTHERN IRELAND SECTION. *General Principles in Vat Dyeing.* J. W. Reidy, Esq. (Messrs. L. B. Holliday & Co. Ltd.). Thompson's Restaurant, Donegall Place, **Belfast.** 7.30 p.m.

Tuesday, 14th February 1956

SCOTTISH SECTION. *The Dyeing and Finishing of 'Terylene' and 'Terylene' Blends.* J. G. Graham, Esq., and Dr. P. T. Standring (I.C.I. 'Terylene' Council). St. Enoch's Hotel, **Glasgow.** 7 p.m.

continued on page xxxiii

Cuprofix
Printing
Colours
SANDOZ
SANDOZ PRODUCTS
LTD · BRADFORD

QF DURINDONE PRINTING BLACKS

BL and TL

vat dyestuffs for fabric printing

- Non-settling pastes with good resistance to drying-out and to frost.
- High rate of fixation.
- Both give prints of excellent light-fastness, suitable for "fast styles", including furnishing and other fabrics that have to withstand prolonged exposure.
- Both give full blacks and attractive medium to pale greys.
- **TL** gives somewhat redder shades than **BL**

Full information freely available on request :

IMPERIAL CHEMICAL INDUSTRIES LIMITED, LONDON, S.W.1

D.428

THE JOURNAL
OF THE
Society of Dyers and Colourists

Volume 71 Number 8 AUGUST 1955 Issued Monthly

Proceedings of the Society

Some Observations on the Dyeing of Wool at High Temperatures

G. A. Coutie, D. R. Lemin, and H. Sagar

Meetings of the Midlands Section held at the College of Technology and Commerce, Leicester, on 6th October 1954, Mr. S. R. Meadows in the chair; and of the Huddersfield Section held at the Co-operative Society Café, Huddersfield, on 18th January 1955, Mr. J. Calvert in the chair

It has been shown that dyeing temperatures above 100°c. can result in the decomposition of a number of wool dyes, primarily as a result of reduction. The full wet fastness properties of wool dyes can be obtained in short times at elevated temperatures. Quantitative tests indicate marked increases in the rate of redistribution of dye at elevated temperatures. Tests on the physical properties of worsted yarns show that excessive degradation of the wool fibre occurs at temperatures above the boil if strongly acid or neutral dyebaths are employed. It has been shown that wet tenacity is a much more sensitive criterion of fibre degradation than dry tenacity. Conditions of time, temperature, and pH are suggested which will yield satisfactory dyeings at elevated temperatures without producing undue fibre degradation.

In recent years there have been considerable advances in the fields of synthetic fibres, dyeing techniques, and machinery. With some of the newer synthetic fibres such as Terylene and Dacron it has been found necessary to develop new dyeing methods in order to achieve satisfactory dyeings in commercially acceptable dyeing times. The use of dyeing temperatures above 100°c. has been found to be advantageous in dyeing such fibres. Because of their unique fibre properties, a number of these newer synthetic fibres have been blended experimentally with wool and other fibres in order to achieve improved fabric properties. It was natural, therefore, for attention to be directed to the suitability of high-temperature dyeing methods for wool. Furthermore, since wool may be satisfactorily dyed in relatively short times at 100°c., it is to be expected that even more rapid dyeing may be achieved at higher temperatures.

The dyeing of textile fibres at temperatures above 100°c. is not new. In 1945 a process was patented in America[1] for the continuous dyeing of piece goods above 100°c. A few years later Steverlynck developed the "static pressure" system for high-temperature dyeing. In the past few years, therefore, the use of high-temperature dyeing has passed from the experimental and development stages to commercial use, and machines for this purpose are now available from a wide range of manufacturers.

A considerable body of experimental data on the high-temperature dyeing of wool has been accumulated, and in some cases conflicting results have been reported. The object of the present work was to undertake a systematic examination of the dyeing of wool at temperatures above 100°c. and to determine the practical value of such processes by reference to— (*a*) reduction in dyeing time, (*b*) effect on the physical properties of wool, and (*c*) effect on dyeing properties. Since in many cases results obtained on a laboratory scale are not reproduced in practice, and to provide information of maximum practical value, as far as possible all experiments have been carried out on a Pegg single-package high-temperature machine of commercial design which works on the Steverlynck "static pressure" system.

Experimental

STABILITY OF WOOL DYES TO DYEING AT ELEVATED TEMPERATURES

It is well known that under normal dyebath conditions certain dyes may be decomposed. This decomposition is in general due to one of two factors. Firstly there is the sensitivity of a number of dyes to hydrolysis in an acid dyebath. Thus, in carpet yarn dyeing use of certain acid red dyes should be avoided, because changes of colour occur during the long boiling times necessary to achieve the desired standard of levelness. The second general cause of dye decomposition is the formation of reducing substances when wool is boiled, which may cause degradation of some dyes. Thus, when dyeing dyes such as Coomassie Navy Blue 2RNS, an addition of sodium or potassium

dichromate is often made to the dyebath in order to prevent the possibility of decomposition due to the presence of reducing substances liberated from the wool during the dyeing process.

Raising the dyeing temperature above 100°C. is likely to result in a more rapid hydrolysis or reduction of dyes than that occurring below this temperature. A number of workers [2] have reported on the decomposition of dye solutions above 100°C. Whilst this will reveal the sensitivity of dyes to hydrolysis, it will yield no information on reduction breakdown, and there do not appear to be any recorded data on the sensitivity of dyes to decomposition in the presence of wool at elevated temperatures.

For this reason, experiments have been carried out to investigate this subject. Dyeings by the normally recommended method have been carried out for (*a*) the normal dyeing time at 100°C. and (*b*) for one hour at 115°C., after which the dyeings have been compared for colour. On the basis of these tests, the dyes examined have been classified as suitable or unsuitable for high-temperature dyeing (Table I).

TABLE I

Suitability of Wool Dyes for Application at Elevated Temperatures

1. LEVEL-DYEING ACID DYES

A. Suitable for High-temperature Dyeing

Lissamine Flavine FF
Quinoline Yellow A
Lissamine Fast Yellow 2G
Tartrazine N
Tartrazine K
Azo Yellow FFN
Citronine Y
Lissamine Fast Yellow AE
Naphthalene Fast Orange 2G
Naphthalene Orange G
Naphthalene Scarlet R
Naphthalene Red J
Lissamine Fast Red 4G
Lissamine Fast Red B
Lissamine Fast Red BG
Azo Geranine 2G
Azo Geranine B
Carmoisine W
Lissamine Rhodamine G
Rhodamine B
Lissamine Rhodamine B
Solway Rubinol R
Lissamine Red 6B
Lissamine Red 7BP
Solway Purple R
Lissamine Violet 10B
Lissamine Blue BF
Solway Blue RN
Lissamine Ultra Sky R
Solway Blue BGA
Solway Ultra Blue B
Solway Blue PFN
Solway Blue BN
Solway Blue A
Solway Blue G
Solway Blue 2G
Indigo Carmine X
Disulphine Blue FFN
Disulphine Blue AN
Disulphine Green B
Naphthalene Green G
Lissamine Green V
Naphthalene Black CW
Naphthalene Blue Black C

B. Decomposed when dyed for 1 hr. at 115°C.

Croceine Scarlet 3B
Naphthalene Scarlet B
Naphthalene Scarlet 4R
Lissamine Fast Red 3G
Carmoisine L
Naphthalene Bordeaux B
Acid Magenta A
Lissamine Violet 2R
Lissamine Violet AV
Lissamine Fast Violet 2B
Lissamine Violet 6BN
Solway Celestol B
Disulphine Blue VN
Lissamine Green BN
Lissamine Green SF
Naphthalene Dark Green A
Lissamine Navy FK
Lissamine Navy Blue FG
Lissamine Fast Navy 2G
Lissamine Navy AN
Lissamine Navy Blue 3G
Naphthalene Black BDV
Naphthalene Black 4BW
Naphthalene Black 12B
Naphthalene Black ESN
Naphthalene Black L

2. ACID MILLING DYES

A. Suitable for High-temperature Dyeing

Coomassie Yellow 7G
Coomassie Yellow 6G
Coomassie Fast Yellow G
Coomassie Yellow R
Coomassie Fast Scarlet 2G
Coomassie Milling Scarlet G
Coomassie Fast Scarlet B
Coomassie Milling Scarlet 5B
Coomassie Red BR
Coomassie Bordeaux B
Coomassie Geranine 2G
Solway Violet BR
Solway Violet BN
Coomassie Violet R
Coomassie Blue BL
Coomassie Blue GL
Coomassie Brilliant Blue R
Coomassie Brilliant Blue FF
Coomassie Ultra Sky SE
Coomassie Brilliant Blue G
Solway Green G
Coomassie Fast Green GN
Coomassie Fast Grey 3G
Carbolan Yellow 4G
Carbolan Yellow 3G
Carbolan Yellow R
Carbolan Crimson B
Carbolan Crimson 3B
Carbolan Violet 2R
Carbolan Brilliant Blue 2R
Carbolan Blue R
Carbolan Blue B
Carbolan Brilliant Green 5G
Carbolan Green G

B. Decomposed when dyed for 1 hr. at 115°C.

Coomassie Fast Orange G
Coomassie Fast Brown R
Coomassie Brown G
Coomassie Red PG
Coomassie Red R
Coomassie Red G
Coomassie Violet 2R
Coomassie Blue RL
Solway Sky Blue B
Coomassie Green T
Coomassie Navy Blue 2RN
Coomassie Navy Blue GN
Coomassie Navy Blue G
Coomassie Navy Blue D
Coomassie Fast Black B
Coomassie Fast Black G
Carbolan Fawn R

3. MORDANT DYES

A. Suitable for High-temperature Dyeing

Solochrome Yellow 2G
Solochrome Yellow 2GN
Solochrome Flavine G
Solochrome Yellow C
Solochromate Fast Orange 2R
Solochrome Brown M
Solochrome Brown RH
Solochromate Fast Red G
Solochromate Fast Red 3G
Solochromate Fast Red 6G
Solochrome Red D
Alizarine Red A
Solochromate Fast Bordeaux BN
Solochromate Fast Violet RL
Solochromate Fast Violet B
Solochrome Brilliant Violet BR
Solochrome Brilliant Violet R
Solochrome Fast Navy 2R
Solway Blue Black B
Solochromate Fast Olive B
Solochrome Black 6BN
Solochrome Black WDFA

B. Decomposed when dyed for 1 hr. at 115°C.

Solochrome Flavine R
Solochrome Orange GR
Solochrome Red ER
Solochrome Brilliant Red 3B
Solochrome Brown EB
Solochrome Brown MG
Solochrome Brown P
Solochrome Brown RG
Solochromate Fast Brown 3G
Solochromate Fast Brown G
Solochrome Violet R
Solochrome Azurine B
Solochromate Fast Blue B
Solochrome Dark Blue B
Solochrome Green V
Solochrome Black PV
Solochrome Black PG

EFFECT OF HIGH-TEMPERATURE DYEING ON WET FASTNESS PROPERTIES

One of the most important questions facing the dyer is that of the fastness properties of any dyeing. Since it has been claimed that the use of temperatures above 100°C. results in decreased dyeing times, it is essential to know whether the fastness

properties of the resultant dyeings are different from those obtained by conventional dyeing methods.

For this reason some preliminary experiments have been carried out on (*a*) Solochrome Azurine BS dyed by both afterchrome and chromate methods, and (*b*) Carbolan Blue BS dyed with 3% ammonium acetate. Dyeings at 1% depth were carried out on 1-lb. samples of 64s quality botany slubbing in the Pegg machine under the following conditions of time and temperature.

The afterchrome dyeings of Solochrome Azurine BS were dyed at 110°C. for 15 and 30 min., and then chromed for 1, 2, 5, and 10 min.; and at 120°C. for 5 and 10 min., and then chromed for 1, 2, and 5 min. A standard dyeing at 100°C. was dyed for 1 hr., and then chromed for 30 min.

With the chromate dyeings of Solochrome Azurine BS and the ammonium acetate dyeings of Carbolan Blue BS, dyeings were carried out for 15, 30, and 60 min. at 110°C. and for 5, 10, and 20 min. at 120°C. The control dyeings at 100°C. were dyed for 1 hr. with both of these dyeing methods.

After being dyed, the samples of wool were dried and then submitted to the following tests—milling, No. 3 and No. 4 wash tests [3]. In addition, potting tests in water at 70°C. for 40 min. and in water at 100°C. for 10 min. were carried out on samples of the dyed material tightly wrapped in pieces of undyed botany serge.

It was found in all cases that the wet fastness properties of the samples dyed at elevated temperatures were equal to those of the dyeings carried out under normal dyebath conditions at 100°C.

EFFECT OF ELEVATED TEMPERATURES ON THE LEVELLING PROPERTIES OF WOOL DYES

Two of the most important factors influencing the production of level dyeings are the rate of adsorption of dye and the levelling (or redistribution) which occurs under normal conditions during the boiling period, and these factors have been the subject of considerable study in recent years. All the literature on high-temperature dyeing is agreed that increase in temperature results in a more rapid adsorption of dye. The information at present available on the migration of wool dyes above 100°C. shows some divergence of opinion, and virtually no quantitative results have been published. Thus whilst it has been reported [2] that dye transfer increases rapidly with increase in temperature, and Drijvers [4] has stated that dyes with a known poor levelness rating at normal temperatures can be used without special precautions for shading at elevated temperatures, Iannarone *et al.* [5] state: "In general, those dyes possessing good levelling properties at 205–208°F. were equally good or better at 225°F. On the other hand, milling dyes which show poor transfer at 205–208°F. exhibit only an insignificant improvement at 225°F.".

Work has been carried out on quantitative lines in an attempt to clarify the position, and some preliminary experiments were carried out on cheeses, commencing dyeing at 95°C. and dyeing for short periods of time using one-way circulation only in an attempt to obtain ring-dyed cheeses. The idea was then to determine the time to obtain level results from a badly unlevel initial dyeing. It was found that even with acid milling dyes, using a dyeing time of only 2 min., the levelness and the penetration were too good for a technique of this type to be satisfactory.

A different approach was therefore examined, and the following technique was eventually developed—

500-g. cheeses were dyed as follows: the dyebath was set at 40°C. with 1% dye and the requisite assistants. The wetted-out cheese was entered, and the temperature raised to 95°C. in 30 min. and held at this value for 60 min., after which the material was rinsed and dried. A composite cheese was then prepared by winding 100 g. of white wool on to a spindle and then winding 100 g. of the dyed wool on top of this.

A levelling bath was then prepared at 95°C. with the requisite assistants for the 200-g. sample, which was entered, and circulation was allowed to proceed from inside to outside only. For levellings at temperatures above 95°C., the package was entered at 95°C., and the temperature raised to the required value in 5 min. and then held there for the necessary time.

After levelling, the yarn was removed and dried. Samples from the originally dyed and originally undyed yarns were removed, and the dye was stripped by Soxhlet extraction with 25% aqueous pyridine and compared colorimetrically in a Spekker absorptiometer with a sample taken from the dyed yarn before levelling.

A number of level-dyeing acid dyes were examined initially. It was found that, under the experimental conditions employed, even at 95°C. levelling was virtually complete in 10–15 min. with—

Naphthalene Orange G
Solway Blue RN
Lissamine Fast Red B
Solway Rubinol R.

Times shorter than these are difficult to control accurately under machine conditions, and attention was therefore turned to acid milling dyes. Tests were carried out on a number of these dyes and on one Ultralan dye, the results being shown in Table II.

EFFECT OF THE DYEING PROCESS ON THE PHYSICAL PROPERTIES OF THE YARN

In the first place cheeses (2/32s worsted yarn, 60s quality) were treated in blank dyebaths containing the following reagents—

(1) 8% Sulphuric acid
(2) 3% Sulphuric acid + 10% Glauber's salt
(3) 3% Acetic acid (30%) + 10% Glauber's salt
(4) 40% Glauber's salt

TABLE II

Effect of Temperature on the Levelling of Wool Dyes

(D = % dye on originally dyed material; U = % dye on originally undyed material)

Dye and Assistant(s)	Time of Levelling (min.)	Temperature of Levelling 95°C. D	95°C. U	110°C. D	110°C. U	120°C. D	120°C. U
1% Coomassie Fast Grey 3GS + 3% acetic acid (30%) + 10% Glauber's salt	20	85·9	14·1	73·3	26·7	58·4	41·6
	40	80·8	19·2	65·5	34·5	55·6	44·4
1% Coomassie Fast Grey 3GS + 40% Glauber's salt	10	—	—	49·4	27·8	43·8	33·3
	20	62·5	17·1	46·2	33·3	40·8	35·6
	40	53·5	24·2	—	—	—	—
1% Carbolan Crimson 3BS + 3% ammonium acetate	20	85·9	14·1	72·5	24·6	60·7	38·8
	40	78·0	22·0	63·5	33·5	54·7	45·3
1% Carbolan Crimson 3BS + 40% Glauber's salt	10	—	—	—	—	51·4	32·1
	20	70·5	16·0	61·0	27·1	46·2	36·3
	35	—	—	58·5	32·4	—	—
1% Coomassie Blue BLS + 3% acetic acid (30%) + 10% Glauber's salt	10	89·2	10·8	66·8	33·2	53·1	46·9
	20	82·8	17·2	59·8	40·2	50·0	50·0
1% Ultralan Blue 2GS + 8% sulphuric acid	10	—	—	65·0	35·0	58·7	40·3
	20	78·0	22·0	56·7	43·3	54·0	46·0
	40	67·2	32·8	—	—	—	—
1% Carbolan Brilliant Green 5GS + 3% ammonium acetate	20	—	—	—	—	62·7	37·3
	40	—	—	72·5	27·5	55·3	44·7
	80	84·3	15·7	62·4	37·6	—	—
	160	77·0	23·0	—	—	—	—
1% Carbolan Brilliant Green 5GS + 40% Glauber's salt	20	—	—	—	—	55·1	44·9
	40	86·2	13·8	61·3	38·7	52·1	47·9
	80	78·3	21·7	55·0	45·0	—	—
1% Carbolan Yellow 4GS + 3% ammonium acetate	20	—	—	75·9	24·1	70·7	29·3
	40	79·8	20·2	67·9	32·1	60·4	39·6
	80	76·1	23·9	—	—	—	—

(5) Afterchrome (3% acetic acid (30%) + 10% Glauber's salt, exhausting with 1% formic acid and chroming with 1% sodium dichromate)

(6) Chromate (2% ammonium sulphate + 1% sodium dichromate).

Three different "dyeing" periods were employed—

(i) 60 min. at 100°C.

(ii) 5 min. at 120°C.

(iii) 15 min. at 120°C.

After dyeing, the material was given a light rinse for 2 min. in cold water and then dried, after which the tensile properties of the yarns were examined—

(*a*) Breaking load, both wet and dry, was measured by means of a single-thread pendulum tester, of capacity 500 g., operating under constant-rate-of-traverse conditions (12 in./min.). In the case of the dry tests all the yarns were conditioned for 24 hr. before testing at 65% R.H. and 21°C. The yarn samples used for the determination of wet strength were immersed initially in water at 100°C., which was then allowed to cool to 21°C. overnight.

Each sample was removed from the water and inserted immediately in the jaws of the tester.

For both wet and dry tests the samples were selected from various positions within each package so as to cover the possibility of variation between outside, inside, and centre.

The positional differences in strength detected were actually small, and were not considered to be significant practically.

(*b*) Yarn elastic recovery was determined by means of a Cambridge Fibre Extensometer, working at a constant rate of loading and unloading (500 g./min.). During the test period the yarn specimens (100 cm.) were extended 2% and the linear recovery from this deformation was measured.

(*c*) Alkali solubility tests, by the method of Harris and Smith [6], were carried out with yarn samples from the cheeses treated at 100°C. and at 120°C. in an attempt to assess fibre damage.

The material (1 g.) was treated in 0·1 N. sodium hydroxide solution, liquor ratio 100 : 1, at 65°C. for 1 hr.

After filtration, washing, drying, and conditioning, the weight of residual fibre was determined.

The results of these tests are contained in Tables III and IV together with the final dyebath pH values.

(*d*) Resistance to abrasion was assessed by means of a Martindale Abrasion Tester, the abradant surface being a crossbred tie lining fabric.

TABLE III

Yarn Properties— Dry

"Dyeing" Method*	Final Dyebath pH	Count	Strength (oz.)	Count × Strength	Extensibility (%)	Alkali Solubility (%)	Elastic Recovery (%)
Untreated wool ...		15·4	9·5	146	14·0	13·2	75·0
95% Confidence limits		±0·4	±0·2	±3	±0·8	±0·2	—
1 (i) ...	2·16	15·2	8·8	134	17·1	46·6	57·6
1 (ii) ...	2·20	15·9	7·7	122	13·4	57·3	58·6
1 (iii) ...	2·18	15·1	7·2	109	11·9	65·5	54·4
2 (i) ...	2·96	15·3	9·6	147	17·7	18·4	59·4
2 (ii) ...	3·00	15·0	9·0	135	16·1	20·2	59·9
2 (iii) ...	2·90	16·0	8·1	130	12·3	22·5	57·5
3 (i) ...	4·84	15·4	9·0	139	13·4	12·9	62·2
3 (ii) ...	4·84	15·0	9·1	136	15·2	13·6	59·6
3 (iii) ...	4·95	15·5	8·1	126	12·7	14·6	57·7
4 (i) ...	6·9	14·9	9·1	136	13·7	11·6	58·8
4 (ii) ...	7·1	15·4	6·7	103	9·1	16·1	55·2
4 (iii) ...	7·1	15·6	6·1	95	8·6	18·2	53·1
5 (i) ...	5·14	15·0	9·3	139	14·1	14·3	60·7
5 (ii) ...	5·30	14·9	8·8	131	14·2	15·2	59·5
5 (iii) ...	5·25	15·2	8·8	134	12·4	16·1	60·6
6 (i) ...	6·45	15·5	9·5	147	16·7	10·9	61·2
6 (ii) ...	6·75	15·1	8·6	130	12·8	10·9	60·4
6 (iii) ...	6·60	15·7	7·8	122	10·6	12·3	56·9
95% Confidence limits		±0·6	±0·6	±10	±2·3	±2·2	—

*See pp. 435–436

TABLE IV

Yarn Properties—Wet

Dyeing Method	Final Dyebath pH	Count	Strength (oz.)	Count × Strength	Extensibility (%)	Alkali Solubility (%)
1 (i) ...	2·16	15·9	7·7	123	41·5	46·6
1 (iii) ...	2·18	14·5	4·4	64	39·2	65·6
4 (i) ...	6·9	15·7	8·9	140	43·0	11·6
4 (iii) ...	7·1	16·2	3·5	57	36·3	18·2
6 (i) ...	6·45	14·8	8·8	130	41·7	10·9
6 (iii) ...	6·60	15·7	6·7	105	40·2	12·3

For this test knitted fabrics (plain knit) were prepared, under controlled conditions, from the treated yarns. Circular patterns were cut from the knitted fabrics and sewn on to a cotton gaberdine backing before mounting in the cups of the tester. In each case the material was abraded to fabric failure, i.e. until a hole appeared. The results are given in Table V.

(*e*) The handle of the treated yarns was assessed subjectively, and the knitted fabrics prepared as above were used for this purpose.

Handle is, of course, an elusive property, but nevertheless a particularly important attribute of a yarn to the garment manufacturer. In assessing this property, therefore, it was considered advisable to approach experts, drawn from different sections of the textile industry, who day by day are called upon to assess and appraise the handle of dyed yarns. A statistically planned test was carried out with 26 subjects, in which pairs of fabrics were assessed, both with and without sight of the fabrics. In each case the subject was asked to select the fabric which, in his opinion, possessed

TABLE V

Resistance to Abrasion— Dry

Dyeing method	1		2		3		4		5		6	
	(i)	(iii)	(i)	(iii)	(i)	(iii)	(i)	(iii)	(i)	(iii)	(i)	(iii)
No. of revolutions to produce holing ...	3,250	1,750	3,750	3,250	5,750	3,750	6,750	1,000	6,750	4,750	7,500	2,500

the best handle. The subjects also ranked the six fabrics prepared from yarns "dyed" at 120°C. for 15 min., in order of handle (1–6, best to worst). The results of these subjective tests are given in Tables VI–VIII.

TABLE VI

Fabric Handle— with Vision

Dyeing Method	Preferences (%) (i)	(ii)	(iii)	Probability
(1)	17·3	43·6	39·1	0·01
(2)	41·0	30·1	28·9	0·40
(3)	43·6	24·4	32·0	0·08
(4)	34·6	32·1	33·3	0·90
(5)	37·8	16·7	45·5	0·001
(6)	53·9	16·0	30·1	0·0001

TABLE VII

Fabric Handle— without Vision

Dyeing Method	Preferences (%) (i)	(ii)	(iii)	Probability
(1)	23·1	36·5	40·4	0·20
(2)	26·3	32·7	41·0	0·25
(3)	40·4	26·3	33·3	0·09
(4)	39·8	26·9	33·3	0·35
(5)	35·9	28·2	35·9	0·40
(6)	46·2	25·6	28·2	0·05

It should be noted that each assessor stated that, for him or her, softness was the primary attribute in judging handle.

TABLE VIII

Fabric Handle— Ranking

Dyeing Method (iii)	Average Score	Ranking Order
(1)	1·62	1
(2)	2·73	2
(3)	3·31	3
(4)	3·75	4
(5)	4·88	6
(6)	4·71	5

In a second series of experiments two dyeing methods only were employed, Method 3 (acetic acid and Glauber's salt) and Method 6 (chromate), for the following reasons—

(i) Methods 1 and 4 (8% sulphuric acid and 40% Glauber's salt (neutral) respectively) produce excessive degradation of the fibre at elevated temperatures.

(ii) It is not considered likely that level-dyeing acid dyes, applied from baths containing sulphuric acid and Glauber's salt (Method 2), will be widely employed at temperatures above the boil, since faster dyes may be applied with comparative ease at these temperatures.

(iii) The chromate method is more convenient than the afterchrome method for high-temperature dyeing.

TABLE IX

Yarn Properties— Dry

Temp. (°C.)	Time (min.)	Count	Strength (oz.)	Count × Strength	Extensibility (%)	Alkali Solubility (%)
	Untreated wool ...	15·4	9·5	146	14·0	13·2
			DYEING METHOD (3)			
100	60	15·6	8·8	137	11·6	10·3
105	15	15·7	8·4	132	12·0	10·7
	30	15·7	8·8	138	11·5	11·0
	60	15·3	8·7	133	11·6	10·8
112	15	15·5	9·3	144	12·4	11·0
	30	15·9	8·7	138	10·5	10·9
	60	16·2	8·4	136	10·4	11·2
120	15	15·3	8·1	124	9·7	11·9
	30	15·4	7·7	119	8·6	12·9
	60	15·8	6·9	109	6·7	16·1
			DYEING METHOD (6)			
100	60	15·5	9·1	141	13·0	9·7
105	15	15·8	8·9	140	12·0	10·2
	30	15·4	9·0	139	12·1	9·6
	60	16·2	8·3	134	11·2	8·9
112	15	15·4	8·8	136	11·7	10·0
	30	15·9	9·0	143	11·9	9·3
	60	16·0	8·5	136	10·5	9·7
120	15	15·2	8·7	132	10·5	9·6
	30	15·3	8·5	130	10·2	9·9
	60	16·0	7·2	115	7·3	13·9
	95% Confidence limits	±0·5	±0·6	±9	±1·0	±0·9

TABLE X

Yarn Properties— Wet

Temp. (°c.)	Time (min.)	Count	Strength (oz.)	Count × Strength	Extensibility (%)	Alkali Solubility (%)
			DYEING METHOD (3)			
100	60	15·6	8·3	129	39·2	10·3
105	15	15·7	8·8	138	40·5	10·7
	30	15·7	8·4	132	37·9	11·0
	60	15·3	8·3	127	39·1	10·8
112	15	15·5	8·6	133	39·6	11·0
	30	15·9	8·0	127	38·4	10·9
	60	16·2	6·8	110	37·3	11·2
120	15	15·3	6·9	106	37·0	11·9
	30	15·4	5·5	85	33·2	12·9
	60	15·8	3·7	59	30·4	16·1
			DYEING METHOD (6)			
100	60	15·5	9·0	139	40·8	9·7
105	15	15·8	10·0	158	41·2	10·2
	30	15·4	8·6	133	39·8	9·6
	60	16·2	8·5	138	39·9	8·9
112	15	15·4	8·6	133	39·9	10·0
	30	15·9	8·0	127	38·7	9·3
	60	16·0	7·0	112	36·6	9·7
120	15	15·2	7·6	115	38·6	9·6
	30	15·3	5·9	90	37·1	9·9
	60	16·0	4·1	66	36·6	13·9
95% Confidence limits		±0·5	±0·7	±11	±2·5	±0·9

Since it has been claimed that glue acts as a protective colloid in minimising fibre damage [7], experiments were included with 5% glue added to the dyebath. Yarn samples were treated at the boil for 60 min. using both methods and also with all combinations of 15, 30, and 60 min. for the three temperatures 105°C., 112°C., and 120°C.

Dry and wet tenacity determinations were carried out on the treated materials using the methods described above. Alkali solubility was also measured.

The results of these tests are contained in Tables IX and X.

Discussion of Results

DYEING PROPERTIES

The data on the stability of wool dyes to dyeing at 115°C. indicated in Table I reveal a wide range of dyes stable under the conditions of test. On the other hand, a number of dyes show marked changes in hue or depth of colour when dyed on wool for 60 min. at 115°C. As a class, the chrome dyes show greater sensitivity to high-temperature dyeing than the acid dyes. It will be clear from this table that in general decomposition follows well defined lines. The dyes listed as unstable are those known to be sensitive to either hydrolysis or reduction, although there are one or two exceptions to this rule. Thus, for example, Azo Geranine 2G, which is sensitive to prolonged boiling in an acid dyebath, is stable when dyed for 60 min. at 115°C.; similarly, Solochrome Brown RH, which is sensitive to reduction and is therefore not recommended for dyeing Ardil protein fibre, is satisfactory for high-temperature dyeing.

From the list of dyes stable when dyed for 60 min. at 115°C., a range may be selected covering all colours for all types of wool dyeing.

From the results of the limited wet fastness tests carried out, all the samples dyed at elevated temperatures possessed equal fastness to those dyed under the normal dyebath conditions at 100°C. Thus, full wet fastness properties can be achieved in extremely short times at elevated temperatures. At 120°C. in the case of the chromate dyeing of Solochrome Azurine B, a dyeing time of 5 min. gives full wet fastness properties. By the after-chrome process, the short dyeing times are even more striking when it is considered that complete chroming appears to have occurred in a time of 1 min. at 110°C or 120°C. Thus, at 120°C. a satisfactory dyeing was obtained in 6 min., compared with a normal minimum time of 90 min. after reaching 100°C. It is clear, therefore, that considerable saving in time can be achieved if the required dyeing properties are obtainable at elevated temperatures, and information on this aspect of the problem is contained in Table II. Before considering these results, it is interesting to note that, under the experimental conditions employed, the levelling of a number of typical level-dyeing acid dyes at 95°C. was so rapid that quantitative tests were virtually impossible to carry out. This indicates the superiority in efficiency of high-temperature dyeing machinery compared with conventional machines.

With the faster types of acid dye, a consideration of the results in Table II shows that, as the temperature of levelling is increased, so does the amount of dye transferred from dyed to originally undyed material also increase. This occurs with all the acid milling dyes when dyed under either acid or neutral conditions, and also with the single Ultralan dye dyed with 8% sulphuric acid. It is also interesting to note that, with the acid dyeings of milling dyes, virtually all the dye is on the fibre, the levelling liquor being virtually colourless; in the case of the neutral dyeings, considerable losses of colour into the liquor occur. Here, however, the sum of the amounts of dye on the originally dyed and on the originally undyed material after levelling approximates very closely to the value for the exhaustion of the particular dyes measured by normal adsorption methods.

The migration of direct cotton dyes on cotton has been shown to follow an approximately hyperbolic relationship [8], and this is also true for level-dyeing acid dyes on wool. In these cases, it has been found possible to evaluate the "time of half-levelling", which may be defined as the time taken for the originally undyed material in a levelling test to adsorb half the dye which it would adsorb at equilibrium, by the use of the following equation—

$$t_{\frac{1}{2}} = \frac{t}{x}(F - x)$$

(F = percentage of dye adsorbed by the originally undyed material at equilibrium; $t_{\frac{1}{2}}$ = time of half-levelling in minutes; and x = percentage of dye adsorbed by the originally undyed material at time t).

From the results in Table II together with a levelling test carried out for a prolonged time to determine the equilibrium value F, the times of half-levelling shown in Table XI have been obtained.

From these results it will be seen that acid milling dyes, when dyed neutral, level more rapidly than under acid conditions, which agrees with accepted dyeing practice. It should be pointed out, however, that at elevated temperatures it is preferable to apply these dyes under weakly acid conditions to obviate fibre damage. The most striking feature of the results in Table XI is the marked drop in times of half-levelling which occurs at 110°C. compared with 95°C., and at 120°C. compared with 110°C. From these figures, at 120°C. levelling occurs, on average, ten times as rapidly as at 95°C., and approximately three times as rapidly as at 110°C.

Thus it is certain that considerable saving of time can be achieved by dyeing wool at elevated temperatures. If, for example, satisfactory dyeings can be obtained in 90 min. at the commercial boil, equally satisfactory results should be obtained in 30 min. at 110°C. or 10 min. at 120°C. It is clear, therefore, that the use of high-temperature dyeing can yield considerable increases in output per machine.

PHYSICAL PROPERTIES

It is essential that any commercial dyeing process should not degrade the material to such an extent that the behaviour of the yarn or fabric during subsequent processing, or during wear, is adversely affected.

Yarn strength depends to a large extent on fibre strength, which in turn is related to, and in fact controlled by, the extent to which the fibre suffers either mechanical damage or chemical attack during wet processing. Measurements of yarn or fibre strength have been used by numerous investigators, therefore, to assess chemical damage to the fibre.

Elöd and his collaborators [9] have studied the effects of acids on the properties of wool, and have suggested that rupture of a relatively small number of peptide bonds results in extensive weakening of the fibres; e.g. these authors claim that when 5% of the peptide bonds are hydrolysed the loss in dry strength is approx. 50% and the loss in wet strength almost 90%. The much higher loss in the case of wet strength can be explained

TABLE XI

Effect of Temperature on Times of Half-levelling of Wool Dyes

Dyeing (1%)	Dyeing Method	Time of Half-levelling (min.) 95°C.	110°C.	120°C.
Coomassie Fast Grey 3GS	3% Acetic acid (30%) ... ... 10% Glauber's salt	56·5	17·8	4·5
Coomassie Fast Grey 3GS	40% Glauber's salt	25·1	3·7	1·8
Carbolan Crimson 3BS	3% Ammonium acetate ...	51	20·2	5·0
Carbolan Crimson 3BS	40% Glauber's salt	34	11·8	3·7
Carbolan Brilliant Green 5GS	3% Ammonium acetate ...	181·5	29·6	5·7
Carbolan Brilliant Green 5GS	40% Glauber's salt	104·5	10·3	2·1
Carbolan Yellow 4GS	3% Ammonium acetate ...	73·7	21·9	12·3
Coomassie Blue BLS	3% Acetic acid (30%) ... ... 10% Glauber's salt	37·2	5·0	0·7
Ultralan Blue 2GS	8% Sulphuric acid	23·3	3·7	2·1

by the proportionately greater effect of main-chain disintegration in the swollen as opposed to the dry state. For this reason wet tenacity is a more sensitive index of fibre degradation than is dry tenacity.

Since wool is susceptible to hydrolysis under both acid and alkaline conditions at the boil, it appeared reasonable to assume that the rate and the extent of the hydrolysis would increase appreciably, under certain conditions, at temperatures above 100°C. This proved to be the case with certain of the dyeing methods employed, notably Method 1 (8% sulphuric acid) and Method 4 (neutral). A considerable drop in both dry and wet strength occurred at 120°C. compared with dyeing at the boil (see Tables III and IV). The damage in the first case (Method 1) was due, in all probability, to peptide chain hydrolysis, since the disulphide bond is not usually broken under such conditions. Peryman [10] has shown that the percentage of disulphide sulphur in the fibre is not seriously reduced by boiling for 3 hr. in acid solutions whose pH is less than 4·7, although the alkali solubility increases considerably. This author, and also Elsworth and Lees [11], report values up to 66·5% for the alkali solubility of wool treated in boiling dilute sulphuric acid of pH 1·5, which corresponds roughly to about 8% by weight on the fibre.

In the present series of experiments very high values for alkali solubility were obtained when using the strongly acid bath, the highest value (65·5%), corresponding to a loss in wet tenacity of 50%, being obtained after treatment at 120°C. for 20 min.

The case of the neutral dyebath (Method 4) is particularly interesting, since appreciable degradation of the fibre, judged by wet and dry tenacity, took place within 5 min. at 120°C. Various authorities have referred to the susceptibility of wool to damage in neutral or near neutral solutions, particularly in the presence of electrolytes. Peryman [10] has drawn attention to the degradation which takes place when wool is treated in solutions of sodium sulphate (pH 6·8 and above) at or near the boil. This degradation is attributed to alkali damage involving disulphide bond breakdown, which increases rapidly with increase of salt concentration and rise of pH.

In the experiments described above, the rate and the extent of degradation in the neutral bath at 120°C. were actually of the same order as those occurring in the strongly acid bath. Dyeing in strongly acid or neutral solutions at 120°C. also produces a marked reduction in the resistance to abrasion, as would be expected from the effect which such processes have on the yarn strength. Although the abrasive action to which the knitted fabrics were subjected is not necessarily representative of all conditions likely to be encountered in wear, the results obtained indicate that a reduced service life is probable when using strongly acid or neutral baths at 120°C.

Reference has been made to the high alkali solubility figures obtained with the fibre treated in the dyebath containing 8% acid. Very much lower values for alkali solubility were obtained from the material treated in the neutral dyebath, although on the basis of strength determinations both yarns were degraded to about the same extent.

In fact, taking the samples treated by the six different dyeing methods in the first series of experiments, there is no evidence of any correlation between alkali solubility and either wet or dry tenacity over the whole range. There is, admittedly, with individual dyeing methods, a rise in alkali solubility with fall in tenacity in each case. Clearly, the alkali solubility test should only be used with discrimination as a means of assessing damage. Its use should be reserved, as pointed out by Elsworth and Lees [11], for those cases in which samples have been subjected to known and comparable treatments. The comparatively low values obtained for alkali solubility with the yarn dyed by the chromate method may have been due to the formation within the fibre of cross-linkages involving chromium atoms [12].

Apart from the effect on yarn strength, treatment under neutral dyeing conditions at 120°C., even for a short period (5 min.), produced a marked reduction in the dry extensibility of the yarn. This is a very undesirable feature, especially in the case of knitting yarns. A value of 8–9% for extensibility probably represents the lower limit for satisfactory knitting with yarns of this type.

In contrast to the yarn extensibility, the elastic recovery properties, under the conditions of measurement employed, were affected to approximately the same extent by all treatments both at the boil and at 120°C.

When the knitted fabrics were examined for handle without vision, the observers detected no appreciable differences between the yarns treated at the boil and those treated under pressure (5 or 15 min. at 120°C.), except with Method 6 (chromate), where a distinct preference was shown for the material dyed at the boil as against that dyed at 120°C., which was found to be harsher than the control yarn. In the case of Method 3 (acetic acid) there was a slight preference for material dyed at the boil (see Table VI). Comparing the decisions with and without vision, it was found that in 280 out of the 468 comparisons the decisions agreed. Statistically this implies that, in the case of the expert, there is strong evidence of some degree of reproducibility, and that on the average the chances are 3 : 2 that the judgments of a person with and without vision will agree. It would be expected, of course, that the tactile sense is of overriding importance in the assessment of handle, but the results obtained imply that vision also plays a part. For example, with vision, the preferences were more pronounced in certain cases. If the three fabrics (i), (ii), and (iii) for any one dyeing method were similar in handle, the expected score in all three cases

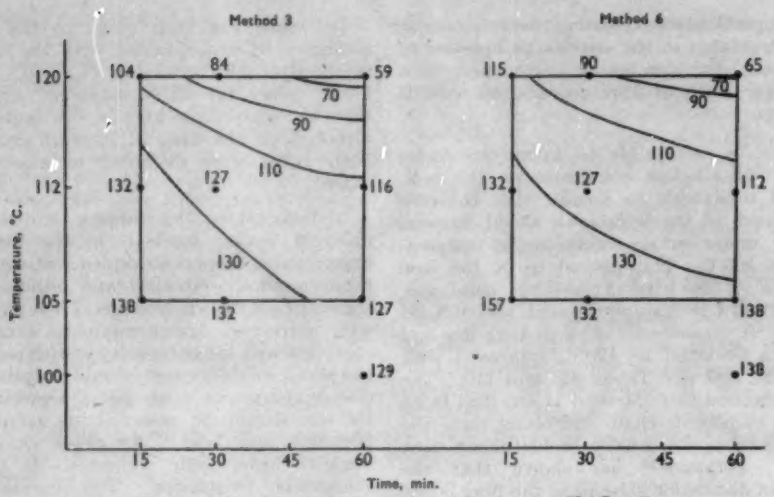


FIG. 1— Effect of Time and Temperature of Treatment on Wet Tenacity

would be 33·3 (Tables VI and VII). In the case of dyeing Method 6 (chromate) the respective values for (i), (ii), and (iii) without vision were 46·2, 25·6, 28·2, while with vision the values were 53·9, 16·0, 30·1. Variations of this order were also obtained with dyeing Methods (1) and (5), implying that with these fabrics the actual appearance of the material had a marked effect on the judgment of the assessors. From the comparison of the effect of reagents on fabric handle, as shown by the order of ranking in Table VIII, there was no doubt that the chrome methods, as might be expected, gave the harshest yarns, and the strongly acid bath the softest yarn; i.e. the highly degraded material was the softest and, in the opinion of the assessors, possessed the most desirable handle.

In the second series of experiments, using Methods 3 (acetic acid) and 6 (chromate), the dry tenacity of the yarn was practically unaffected by dyeing at 105°C. and 112°C. as compared with material dyed at the boil. At 120°C., however, there was a reduction of 20%, rising to over 50% after 60 min. (Tables IX and X). The experimental data obtained when glue was added to the dyebath showed that this colloid does not minimise fibre damage as measured by either wet or dry tenacity. The results confirm those obtained in the first series of experiments, and indicate clearly that wet strength is affected to a greater extent than dry strength by dyeing at elevated temperatures. It is possible that wet abrasion resistance will likewise be affected to a greater extent than the dry. For convenience, the effects of time and temperature for both dyeing methods have been presented in the form of contours, from which it is possible to assess quickly the extent to which these variables affect the wet tenacity of the yarn (Fig. 1). With dyeing Method 3 (acetic acid), for

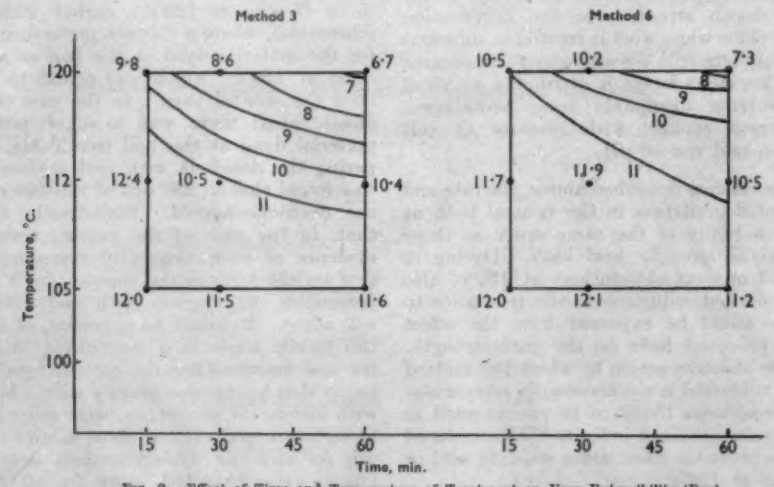


FIG. 2— Effect of Time and Temperature of Treatment on Yarn Extensibility (Dry)

example, the wet tenacity would be reduced by approx. 20% if the yarn were treated under the following conditions—(*a*) 15 min. at 120°C., (*b*) 30 min. at 115°C., or (*c*) 60 min. at 113°C.

Yarn extensibility, both wet and dry, was reduced significantly by dyeing for periods beyond 15 min. at 120°C. using both methods. In view of the importance of yarn extensibility, particularly during knitting operations, the effect of time and temperature on this property has also been illustrated in the form of contours (Fig. 2). For both dyeing methods an increase of 5°C. has a greater effect on dry extensibility than an increase in dyeing time of 15 min.

With dyeing Method 3 (acetic acid) there was an appreciable rise in alkali solubility only after treatment for 60 min. at 120°C. In the other method (chromate) low values for alkali solubility were obtained; e.g. after treatment for 60 min. at 120°C. there was no difference between the alkali solubility of the treated wool and that of the control sample (Table IX).

Using either method, i.e. acetic acid or chromate, little or no damage appears to be caused if the temperature is kept at 112°C. for dyeing periods up to 60 min. It is possible that in the case of chromate dyeings a somewhat harsher yarn may be produced than when dyeing at the boil.

Conclusions

The results obtained indicate not only that the high-temperature dyeing of wool is a practical proposition, but also that it possesses advantages compared with normal dyeing procedures, provided that certain precautions are taken.

The use of strongly acid (pH 2) or neutral dyebaths should be avoided, the preferred limits of pH being 3 and 6·5. In order to avoid undue degradation of the wool fibre, the maximum dyeing temperature should not exceed 110°C., and the time of dyeing at this temperature should be restricted to a maximum of one hour. Under these conditions, avoiding the use of dyes sensitive to decomposition, satisfactory dyeings of acid and chrome dyes can be obtained.

* * *

The authors are indebted to the Hosiery and Allied Trades Research Association for the preparation of the knitted fabrics used in the handle tests and to Mr. W. A. Dutton for helpful discussion in connection with these tests.

DYEHOUSE LABORATORIES
IMPERIAL CHEMICAL INDUSTRIES LTD.
DYESTUFFS DIVISION
HEXAGON HOUSE
MANCHESTER 9

(*MS. received 28th January 1955*)

References

[1] Walter, *USP* 2,387,200.
[2] Royer, Zimmerman, Walter, and Robinson, *Text. Research J.*, **18**, 598 (1948);
AATCC Philadelphia Section, *Amer. Dyestuff Rep.*, **38**, P 9 (1949).
[3] *Second Report of the Fastness Tests Committee* (Bradford: Society of Dyers and Colourists, 2nd edition 1950).
[4] Drijvers, *Amer. Dyestuff Rep.*, **41**, 533 (1952).
[5] Iannarone, Clapham, and Thomas, *ibid.*, **42**, P 666 (1953).
[6] Harris and Smith, *Bur. Stand. J. Res.*, **15**, 63 (1935).
[7] Chwala, *Textilhilfsmittel* (Vienna: Springer Verlag 1939), p. 367.
[8] Lemin, Vickers, and Vickerstaff, J.S.D.C., **62**, 132 (1946).
[9] Elöd, Nowotny, and Zahn, *Kolloid-Z.*, **93**, 50 (1940).
[10] Peryman, J.S.D.C., **70**, 83 (1954).
[11] Elsworth and Loes, *ibid.*, **68**, 207 (1952).
[12] Race, Rowe, and Speakman, *ibid.*, **54**, 159 (1938).

The Dyeing of Textile Fibres above 100°C.

J. A. FOWLER

Meeting of the London Section held in the Rooms of the Royal Society, Burlington House, London W.1, on 3rd December 1954, Dr. H. W. Ellis in the chair

The application is described of high-temperature techniques, i.e. above 100°C., in the dyeing of a variety of textile fibres. Particular mention is made of the application of direct and vat dyes to cellulosic fibres, of acid and chrome dyes to wool, and of disperse and azoic dyes to Terylene polyester fibre. Reference is also made to the machinery available and the advantages to be gained by the use of high-temperature techniques.

Introduction

The dyeing of textile fibres at temperatures above 100°C. has been one of the major post-war advances in dyeing technology. Because of ever rising labour and fuel costs, the technique has aroused particular interest as offering more rapid and surer dyeing methods for the established fibres—cotton, viscose rayon, and wool; the advent of the hydrophobic synthetic fibres such as Terylene polyester fibre with their attendant problem of dyeability at temperatures below 100°C. has also stimulated this interest in high temperatures. It seems useful, therefore, now that suitable dyeing machinery is commercially available, to review very briefly the possibilities of the process for a variety of textile fibres.

Brief reference will be made to the machinery used, and to the application and advantages of the technique in the dyeing of (*a*) cellulosic fibres, (*b*) wool, and (*c*) synthetic fibres (restricted to Terylene for reasons of space).

In the last decade or so, considerable advances have been made in piece-dyeing methods, and the wider employment of padding techniques, including continuous dyeing methods, has given a means of overcoming many of the quality and production problems of piece-dyeing, particularly of cellulosic fibres. For this reason, the high-temperature dyeing technique is of greater interest in the dyeing of cellulosic yarns in the form of wound packages, and only brief reference will be made to piece-goods.

The problem of dyeability with many of the synthetic fibres is not necessarily soluble by piece-padding methods, and in these cases there is a potential field of interest for dyeing both yarn packages and piece-goods above 100°C.

Before proceeding to a consideration of the mechanics of the process, there is a certain confusion of nomenclature to be cleared up: the use of temperatures above 100°C. has been referred to as both "high-temperature" and "high-pressure" dyeing. I feel that the latter term is misleading, and in this paper the term *high-temperature* is used throughout to signify dyeing above 100°C.

Dyeing Machinery

A. PACKAGE-DYEING EQUIPMENT

Package or "machine" dyeing equipment has long been in use in the textile trade. Much has been said elsewhere on the virtues of a system whereby the dye liquor is circulated through the stationary goods compared with the older methods of moving the material in a more or less static dye liquor.

Package dyeing, whilst demanding a higher degree of skill and patience on the part of the package winder, the dyer, and the dyehouse maintenance engineer, has many advantages in respect of lessened handling of the goods, savings in steam, dyes, and chemicals, increased production, a saving (in the case of yarn) in rewinding charges, and hence lower dyeing costs. Package dyeing has thus found increasing favour in the dyeing trade, in spite of the initial higher capital cost of the equipment compared with older methods of dyeing.

It is well known that with many classes of dyes the production of level results is facilitated by the use of the maximum possible dyeing temperatures. In the case of the synthetic fibres, too, high temperatures increase very rapidly the diffusion of the dyes within the fibre and hence the exhaustion obtained with many dyes in an acceptable dyeing period. However, open package-dyeing equipment suffers from the limitation that water, the usual vehicle for dye application, boils at 100°C. under atmospheric pressure. In consequence of this, with the centrifugal types of pump normally used in package-dyeing equipment, 90–95°C. often represents the maximum temperature obtainable without a marked falling off in the efficiency of the pump.

The possibility of dyeing textile fibres above 100°C. and the behaviour of the various classes of dyes were first explored in the laboratory by various American workers [1,2], who demonstrated the great advantages to be obtained. The process was, however, first operated successfully on a commercial scale by Steverlynck and his co-worker Drijvers in Belgium, using a process now known as the Steverlynck Static Process [3,4].

Since the publication of this work by Drijvers [5], machinery capable of package-dyeing textile fibres above 100°C., both in the laboratory and in bulk, is now available from a number of textile machinery manufacturers.

High-temperature package-dyeing machines now available have many fundamental points of design in common: they are pressure vessels and therefore are of more robust construction than the normal type of "open" machine; they are more complicated mechanically and are therefore more expensive. The necessary static pressure within the machine can be obtained by the introduction of compressed air or gas, e.g. nitrogen, into the system. The normal method of making additions to the dye liquor in, and of sampling dye liquors from, an expansion tank which is open to the atmosphere, and in direct contact with the dyeing chamber, cannot be adopted; additions to, or sampling of liquors from, the system whilst under pressure are made possible by special valve arrangements. Lastly, means of rapid heating, and cooling, of the processing liquors are essential.

Finally, experiments have shown that care spent in packing the machine, or in winding and mounting the packages, is amply repaid. As in the normal types of machines, packs and packages of irregular density, or packages which are incorrectly mounted in the machine, will permit channelling of the dye liquors and so lead to poor results.

B. PIECE-DYEING EQUIPMENT

Open-width pressure kiers for preparing cotton piece-goods have been established in the textile trade for some time. By an extension of these, it is now possible to obtain pressurised jigs capable of dyeing fabrics above 100°C. Such jigs have not as yet found any extensive use in the trade, although they offer a method of dyeing dark colours on synthetic fibres such as Terylene in a reasonable dyeing period without resorting to carrier dyeing techniques.

A second, and entirely novel type of machine is the Barotor (DuP) [6]; again, this machine is more obviously suited to synthetic fibres. A further development in the dyeing of synthetic fibres is the Thermosol (DuP) process [7], in which the dye is applied by padding, the cloth dried, and the dye then fixed in a few seconds at 200–220°C.

In the case of wool, a process for continuously dyeing wool piece-goods above 100°C. was patented in America in 1945 [8].

It is now proposed to consider the various fibres and classes of dyes in detail; the points to which it is proposed to pay particular attention are—

(*a*) Stability of the dyes

(*b*) Effect of the high temperature on the fibre

(*c*) Levelling

(*d*) High-temperature dyeing methods.

Cellulosic Fibres

The classes of dyes of most interest for high-temperature application to cellulosic fibres are direct, vat, and, to a lesser extent, sulphur dyes.

1. DIRECT COTTON DYES

(*a*) *Stability of the Dyes*

The tendency of direct dyes to decompose at or above the boil has been studied by numerous investigators, e.g. Ashpole *et al.*[9], Armfield[10], Butterworth[11], Drijvers[5], and Herrmann[12]; the decomposition is caused by the reducing conditions set up by cellulose in the presence of alkali, and provided such reducing conditions are avoided, the majority of direct dyes are suitable for use at 100–120°C. The range of ICI direct dyes has recently been examined[11], and can be classified into three groups according to their stability in the presence of viscose rayon (Table I). It is noteworthy that, out of 114 dyes examined, only 13 fall into Group III. The bulk of the dyes fall into Group II.

TABLE I

Stability of Direct Dyes on Viscose Rayon at 120–130°C.

GROUP I

Dyes stable when dyed at high temperatures (120–130°C.) under more or less neutral conditions and also showing considerable resistance under alkaline conditions

GROUP II

Dyes not unduly affected by high temperatures in the absence of alkali but completely destroyed under alkaline conditions

GROUP III

Dyes decomposed under both neutral and alkaline conditions

Free alkali can, of course, be introduced into the system via the dyes themselves, auxiliary products used in dyeing, or the material being dyed, or it may arise during processing when certain softened waters are used. It is important to realise that at high temperatures, viz. 120–130°C., this decomposition can be very rapid.

The exclusion of free alkali from the dyeing system is not always possible, and the difficulty may be overcome by the addition to the dyebath of buffers, such as ammonium sulphate.

Where decomposition of the dye is prevented, the normal fastness properties associated with the dye are to be expected.

(*b*) *Effect on the Fibre*

Our experiments have indicated that cellulosic fibres, dyed with direct dyes at 120°C., should suffer no significant loss in strength.

(*c*) *Levelling*

Direct dyes on cellulosic fibres have been classified by the Society according to their behaviour at the boil[13]. On cotton little difficulty is experienced in the application of all classes to packages; but on viscose rayon packages, particularly filament cakes, some of the Class B and Class C dyes give rise to difficulties in practice. The ease of migration of all classes is materially improved by raising the temperature to 120°C.; it is further noteworthy that the migration is much reduced in the presence of electrolyte, which indicates the necessity for exercising the normal control over salt additions during the dyeing operation, even at elevated temperatures.

(*d*) *Dyeing Methods*

(i) Viscose Rayon

The dyeing of viscose rayon filament cakes offers the most striking example of the value of dyeing cellulosic fibres with direct dyes at high temperatures. Filament cakes are the most difficult of all viscose rayon packages to dye. In bulk, dyeing times of 4–6 hr. at 95–100°C. are not uncommon with some Class B and Class C direct dyes; indeed, some Class C dyes do not necessarily give level results on cakes in practice even in 6 hr.

The use of elevated dyeing temperatures opens up an entirely new approach; from experiments carried out in our laboratories and from bulk experience, it is evident that dyeing times can be very significantly reduced by the use of temperatures above 100°C. The time and the temperature required will vary more or less in accordance with the Society's dyeing classification[13], although finer-denier yarns or the dyeing of pale shades, for instance, will naturally mean an increase in the technical difficulty.

In laboratory experiments with viscose rayon cakes (150 denier, 27 filament, bright), Class B and Class C dyes have been dyed level in dyeing times of 1–1½ hr. at 120°C., gradual additions of salt being made at 120°C. to promote exhaustion. With Class A dyes, dyeing temperatures of 105–110°C. are generally sufficient, and it is necessary, after the addition of salt, to cool to 85–90°C. in order to obtain an economic degree of exhaustion. Cooling back is unnecessary with most Class B and Class C dyes.

The normal precautions to be taken when dyeing in conventional cake-dyeing equipment will still apply—cakes must be selected so as to fit satisfactorily on the carriers or holders when wet; salt additions necessary for obtaining economic exhaustion are preferably made gradually by a drip-feed mechanism; the use of concentrated brands of dyes has definite advantages in controlling the electrolyte content of the bath. Finally, the use of non-ionic levelling agents, e.g. Dispersol VL, is of value, particularly in the dyeing of pale colours with dyes of Classes B and C.

In the case of viscose rayon staple fibre packages, e.g. cheeses, cones, warp-beams, or packed hanks and loose stock, the advantages of temperatures above 100°C. are less obvious, because such packs and packages are in any case easier to dye than filament cakes. High temperatures are likely to be valuable in the dyeing of staple fibre warp-beams, where difficulties sometimes arise, or in cases where highly twisted or multi-ply yarns are concerned.

(ii) Cotton

The observations made in the foregoing will serve equally well for cotton also. Grey cotton, by virtue of the reducing substances it contains, is likely to promote reduction of the dyes, and ammonium sulphate should be used in the dyebath as a precaution.

However, in the main, satisfactory levelness and penetration of cotton yarn packs and packages can be obtained by ordinary dyeing methods in reasonable dyeing times, and it is doubtful whether there is much to be gained by the use of temperatures above 100°C.

(iii) Bast Fibres

The use of high-temperature dyeing equipment also opens up interesting possibilities in the dyeing of the cheaper bast fibres, e.g. jute, in package form.

Summary

Summarising the above, the use of high temperatures has very real advantages in the dyeing of viscose rayon filament cakes from the point of view of the saving of dyeing time and the quality of the results obtained. With viscose rayon staple fibre packages and cotton, the advantages are less striking, but in many cases the use of high temperatures appears worthwhile. Under the conditions described, the majority of direct dyes are suitable for the process and produce dyed fibres of satisfactory colour, fastness properties, and physical characteristics.

2. VAT DYES

(*a*) *Stability of the Dyes*

The problem of the stability of leuco vat dyes at high temperatures is much more serious than is that of the stability of direct dyes. Whilst a number of leuco vat dyes are reasonably stable at 70–80°C., temperatures of the order of 120°C. diminish this number considerably.

TABLE II

Vat Dyes suitable for the Leuco Dyeing of Cotton and Viscose Rayon at 120–130°C.

Caledon Yellow 5G
Caledon Yellow GN
Caledon Golden Yellow GK
Caledon Gold Orange GN
Caledon Red 2G
Caledon Red BN
Caledon Violet XBN
Caledon Dark Blue G
Caledon Dark Blue 2R
Caledon Dark Blue BM
Caledon Green BN
Caledon Green G
Caledon Green 2G
Caledon Green 7G
Caledon Green RC
Caledon Jade Green 3B
Caledon Jade Green XBN
Caledon Jade Green XN
Caledon Olive Green B
Caledon Black 2BM
Caledon Black NB

Decomposition of the leuco vat dye may be attributed to a number of causes, e.g. hydrolysis of labile groupings, dehalogenation, molecular rearrangement, and over-reduction. On the basis of experiments on cotton and viscose rayon, the Caledon dyes listed in Table II have been found to be sufficiently stable for application at temperatures of the order of 120–130°C. It is interesting that they include practically all those dyes which would be expected from purely chemical considerations to be stable.

A study of Table II will reveal two obvious colour gaps— bright blue and brown. Most bright blues are related chemically to indanthrone and in the leuco state are unstable to varying degrees at temperatures above 60°C.; the Caledon Browns, whilst more stable than the indanthrone blues, fairly readily undergo hydrolysis even at 100°C.

Numerous substances have been proposed as inhibitors to minimise the degradation of leuco vat dyes, particularly in the indanthrone blue series. Glucose and dextrin[14] have been suggested, and recently the American Cyanamid Co. has patented the use of a number of compounds[15], including inorganic nitrites, chlorates, and hydroxylamine; more recently still, cobalt salts have been included[16].

Such inhibitors are reasonably effective, although in some cases difficulties can arise in practice because of increased consumption of sodium hydrosulphite. Recently triethanolamine[17] has been suggested, in conjunction with sodium nitrite, to overcome this difficulty.

With dyes which are stable at the temperatures used, the normal fastness properties are to be expected.

(*b*) *Effect on the Fibre*

The use of temperatures above 100°C. is common in the preparatory processes for cotton, and it is not to be expected that leuco vat dyeing at 100–120°C. would impair the strength of this fibre; this was confirmed by tests made on mercerised cotton yarn. In the case of viscose rayon the fibre is much more liable to damage by alkali; experiments in the laboratory cheese-dyeing machine, however, indicated that the process was less severe than anticipated (Table III).

TABLE III

Effect of Caustic Soda on Viscose Rayon at High Temperature

(Viscose rayon staple fibre yarn treated in cheese form in liquor containing 15 ml. of caustic soda (68°Tw.) and 5 g. of sodium hydrosulphite per litre)

Temp. (°C.)	Time (min.)	Fluidity
	Untreated yarn ...	8·6
100	30	8·5
	60	9·1
	120	9·7
110	30	8·6
	60	8·9
	120	9·0
120	30	8·4
	60	8·9
	120	9·1

(c) *Levelling*

Leuco vat dyes show a marked increase in their ease of migration as the temperature is raised. Use is often made of this in conventional dyeing processes, and temperatures of 70–80°C. are by no means uncommon in the dyeing of cellulosic fibres with leuco vat dyes; particular use is made in the dyeing of the strong-alkali Method 1 vat dyes on cotton and with all classes of vat dyes on viscose rayon.

In the case of viscose rayon, the ease of migration is less than is the case with unmercerised cotton, and the leuco dyes vary quite considerably among themselves. White-skein levelling tests on viscose rayon staple fibre yarn indicated that the dyes could be classified according to their ease of migration (Table IV).

TABLE IV

Ease of Migration of Leuco Vat Dyes on Viscose Rayon

(White-skein levelling tests for 30 min.; staple-fibre yarn, 35/1 counts (cotton-spun))

(i) LEVEL AT 100°C.
Caledon Yellow 5G
Caledon Yellow GN
Caledon Golden Yellow GK
Caledon Gold Orange GN
Caledon Red 2G
Caledon Red BN
Caledon Violet XBN
Caledon Green G
Caledon Green 2G
Caledon Green RC
Caledon Jade Green XBN
Caledon Jade Green XN

(ii) LEVEL AT 100–120°C.
Caledon Jade Green 3B
Caledon Olive Green B
Caledon Dark Blue G

(iii) NOT LEVEL AT 120°C.
Caledon Dark Blue 2R
Caledon Dark Blue BM
Caledon Green BN
Caledon Green 7G
Caledon Black 2BM
Caledon Black NB

(d) *Dyeing Methods*

(i) Cotton

The difficulties in the package dyeing of cotton with vat dyes are generally associated with Method 1 dyes, and it is common practice to start dyeing at 20–30°C. and then raise the temperature to the normal dyeing temperature, or even to 70–80°C. if the dye is stable. Blackburn and Fox[18] demonstrated, however, that the difference in exhaustion between the temperature of maximum "affinity" (40–60°C. for Method 1 dyes) and 20°C. is very much less than the difference between the temperature of maximum "affinity" and 120°C.

It is therefore logical to assume that a greater degree of levelness is to be expected by starting the dyeing at the high temperature and then cooling, than by the normal temperature-rise method. This was in fact shown to be correct, and with 20/2 bleached cotton yarn perfectly level cheeses, free from white "cross-overs", were obtained with Method 1 vat dyes in dyeing times of 45–60 min., the dyeing being started at 120°C. and cooled gradually to 50°C.

Equally satisfactory results were obtained on 36/3 grey mercerised cotton sewing yarn in tightly wound cheeses by a similar technique. Parallel dyeings by the normal leuco process showed "cross-overs" and relatively poor penetration.

However, it must be admitted that with many cotton packages, and with many vat dyes, perfectly satisfactory results can be obtained by normal dyeing methods, viz. at 50–60°C. High temperatures, viz. 100–120°C., seem likely to prove more valuable for difficult yarns or packages, e.g. those constructed from fine-count, highly twisted, multi-ply, or mercerised yarns, or for hard packages. Even for packages where no particular difficulties are being encountered by the normal methods, the high-temperature machine, capable of very satisfactory operation at 90–100°C., is likely to be valuable as an insurance against faulty dyeings, and will perhaps show a small but worthwhile saving in dyeing time.

The use of Dispersol VL, in the normal way, has been found to be valuable at 100–120°C., particularly with rapidly dyeing dyes sensitive to Dispersol VL.

(ii) Viscose Rayon

With viscose rayon the picture is a little different. Whilst cotton packages are normally dyed by leuco dyeing techniques, in the case of viscose rayon the fibre swells to a much greater extent when wetted and the package is in consequence less easily permeated by the dye-liquors; this, coupled with the lower levelling power of leuco vat dyes on viscose rayon, means that for the production of satisfactory results with filament and staple viscose rayon packages recourse has generally to be had to prepigmentation techniques, e.g. the Abbot–Cox process. Prepigmentation techniques require some care in their operation, and for that reason a leuco dyeing process which gave equally satisfactory results would be an interesting achievement.

Our experiments have indicated that viscose rayon staple fibre packages can in fact be dyed to an acceptable degree of levelness by a leuco dyeing technique, using temperatures of the order of 100–120°C. The actual dyeing temperature is selected according to the migration characteristics of the leuco vat dyes in question, i.e. on the basis of Table IV. Unnecessarily high temperatures should be avoided, as they increase the possibility of degrading the dyes and/or the fibre itself. In addition, in our laboratory cheese-dyeing experiments at high temperatures, there is a tendency for the cheeses to shrink in length, and this is likely to be more marked at 120°C. than at, say, 105°C.

With filament cakes, the production of satisfactory results by leuco dyeing at high temperatures is more difficult than with staple fibre packages, mainly because of the resistance to liquor flow offered by a wet, swollen cake. Where high-

temperature dyeing plant is available, the use of a conventional prepigmentation process, e.g. the Abbot–Cox process, followed by reduction at 100–120°C., is likely to be of value.

Summary

The use of high temperatures, viz. 100–120°C., in the application of leuco vat dyes to cellulosic fibres has advantages in respect of the improved levelling obtainable. A limiting factor is the somewhat restricted choice of dyes and, in the case of viscose rayon, the danger of degrading the fibre; however, the process is clearly of interest to package dyers where difficulties are being encountered with normal methods.

3. SULPHUR DYES

From work reported by Senior [19], sulphur dyes are not unduly affected by temperatures of 100–120°C. The main advantage which accrues from the use of high temperatures with sulphur dyes is the saving in time of dyeing— times of one-sixth of the normal dyeing period have been reported [19]. This shortened time of dyeing has obvious advantages from the points of view of economics and also the condition of the material after dyeing, the latter being particularly evident with viscose rayon.

Wool

In the case of wool, the high-temperature technique is of interest in the application of acid, acid milling, and chrome dyes.

ACID, ACID MILLING, AND CHROME DYES

(*a*) *Stability of the Dyes*

It is well established that, even under normal conditions of application, certain wool dyes can be decomposed. The effect may be one of hydrolysis in an acid bath or of reduction by the reducing substances formed when wool is treated in boiling liquors.

At temperatures above 100°C. these effects of hydrolysis or reduction become more marked; whilst earlier workers [1,2] had studied the sensitivity of the dyes to hydrolysis in solution above 100°C., there appear to be no recorded data on degradation due to reduction. For this reason experiments were carried out on a large number of dyes, in the presence of the wool fibre, for (*a*) the normal dyeing time at 100°C. and (*b*) one hour at 115°C. From these it was found that a considerable number of dyes are stable under high-temperature conditions [20].

Fastness tests carried out on selected acid and chrome dyes show that the normal wet fastness properties can be attained in very short dyeing times at elevated temperatures. As an example, with Solochrome Azurine BS (*C.I.* 720) dyed by the afterchrome process, a dyeing time of 5 min. at 120°C. and a chroming time of 1 min. at 110°C. or 120°C. are sufficient to produce a satisfactory dyeing as far as wet fastness is concerned. This total time of 6 min. at 120°C. should be compared with a normal minimum total dyeing time of 90 min. after reaching 100°C.

(*b*) *Effect on the Fibre*

Interesting observations on the effect of high-temperature dyeing processes on the wool fibre were also made by Lemin *et al.*[20]. A comprehensive series of measurements on the various physical characteristics of the fibre showed that strongly acid (pH 2) or neutral dyebaths should be avoided and that the temperature should preferably not exceed 112°C. in order to avoid damage to the fibre. At this temperature, dyeing times of 20–30 min. are required for satisfactorily level results with acid and chrome dyes.

(*c*) *Levelling*

The published data [1,2,5,21] are somewhat at variance on the degree of levelling to be expected, and for this reason the quantitative work recently carried out and reported by Lemin *et al.*[20] is particularly interesting.

The method adopted was virtually a white-skein levelling test at the various temperatures, in cheese form. The results can conveniently be expressed as "times of half-levelling", which may be interpreted as the time taken for the originally undyed material in a levelling test to absorb half the dye which it would absorb at equilibrium.

The most striking feature emerging from these results is the progressive drop in times of half-levelling which occurs as the temperature is raised: with the dyes examined, on the average, levelling occurs ten times as rapidly at 120°C. as at 95°C., and approximately three times as rapidly as at 110°C. Thus, if satisfactory dyeings can be obtained at 95°C. in, say, 90 min., then equally satisfactory results should be attained in 30–40 min. at 110°C. and 10–15 min. at 120°C.

Summary

The advantages of the high-temperature dyeing of wool lie not only in the much increased levelling to be obtained with acid milling and chrome dyes, but also in the much shorter dyeing times necessary, which in turn mean greater productivity. Suitable precautions must be taken, i.e. pH and temperature control and the selection of stable dyes, if dyed materials of satisfactory properties are to be obtained.

Synthetic Fibres

DISPERSE DYES ON TERYLENE POLYESTER FIBRE

Of all the synthetic fibres, Terylene offers the most interesting possibilities where dyeing above 100°C. is concerned. The problem is, however, a little different from those of the fibres which have been discussed so far.

Of the available fibres, Terylene is chemically most similar to cellulose acetate, but the main difference between the two fibres (apart from physical properties) lies in the resistance of Terylene to dyeing. Waters [22] has shown that Terylene contains an adequate number of sites for the absorption of disperse dyes, but that the rate of diffusion of such dyes into Terylene is about 0·2% of that into cellulose acetate or nylon fibres.

This means that under normal dyeing conditions, i.e. at 85–100°C., the exhaustion obtained in a practicable dyeing time is too low to permit of other than pale and medium depths being obtained, using selected disperse dyes. However, the rate of diffusion of disperse dyes into Terylene increases rapidly with temperature, and at 120°C. heavy dyeings may be obtained with many dyes. An immediate advantage of dyeing above 100°C. is therefore the production of heavy depths in a reasonable dyeing time without the expense and difficulty of carriers: in fact, at 120°C. we are able to utilise the whole capacity of the fibre for absorbing disperse dyes.

Cross-sections of fibres dyed at 85°C. show marked ring-dyeing, whilst at 120°C. the dye has penetrated through the fibre.

A further useful advantage of dyeing at these high temperatures is the improved levelling obtained.

In the case of disperse dyes on Terylene, the questions of dye and fibre stability can be fairly simply dismissed. All the ICI disperse dyes of interest for Terylene that are on the market at present are sufficiently stable under the recommended conditions of application. Under these conditions also, the process is without significant effect on the fibre itself, provided alkalis are excluded from the processing liquors.

The wet fastness properties of disperse dyes dyed at high temperature on Terylene are slightly better than those of similar dyeings produced with a carrier. The light fastness of Terylene dyed at high temperature is approximately the same as when dyed at atmospheric pressure using a carrier such as Tumescal D, and rather higher than when dyed with the aid of Tumescal OP.

Dyeing Methods

When filament yarn is dyed in cheese form it is necessary to relax the yarn before dyeing to prevent shrinkage and the resulting formation of hard packages through which the dye liquor is unable to circulate effectively. This relaxing treatment can be effected by twist-setting in steam, or by winding the yarn on to collapsible spiral holders or cardboard cones, relaxing in water or steam, and then rewinding on cheeses for dyeing; the temperature of the relaxing treatment must be at least that at which dyeing is carried out. The treatment is not required for staple fibre packages.

Before dyeing, the packages should be scoured. For the actual dyeing operation, Lissapol C has been found the most suitable dispersing agent for disperse dyes, a concentration of 0·5–2·0 g./litre being sufficient. It is best to start dyeing at 60–70°C., raise the temperature to 120–130°C., and continue for 30–45 min. at this temperature; in the case of very heavy dyeings, e.g. blacks, it is advisable to add the dyes portionwise. At the completion of dyeing, the yarns are given a light "soaping", or in some cases with dark colours a "reduction–clearing" process may be advisable.

The object of the reduction–clearing treatment is the improvement of the rubbing and wet fastness properties of the dyed fibre. The process is carried out in a bath of the following composition—

Caustic soda (68°Tw.) ...	6 parts
Sodium hydrosulphite ...	2 parts
Lissolamine A 50% Paste ...	2 parts
Water	1000 parts

The treatment is commenced cold; the temperature is then raised to 70°C. and maintained at this level for 10 min., after which the goods are thoroughly washed off before the final "soaping" treatment.

It is perhaps worth noting at this stage that in many cases little, if any, saving in time of processing results from the high-temperature dyeing method: the advantages reside rather in the convenience of the process and the more economic use of dyes.

In the case of piece-dyeing at temperatures above 100°C., similar considerations will apply, although, obviously, filament fabrics must be suitably set before dyeing.

AZOIC DYES ON TERYLENE POLYESTER FIBRE

The azoic process for Terylene involves simultaneous application of coupling component and base as a dispersion, followed by diazotisation on the fibre under special conditions, at 85°C., so as to form the pigment within the fibre. The process is particularly valuable for deep reds, maroons, navies, and blacks of good fastness.

At temperatures up to 100°C., the coupling components which can be absorbed by the fibre have relatively small molecules, e.g. Brentosyn BB (free 2:3-hydroxynaphthoic acid): the Brenthols, in the form of their free arylides, are too large and diffuse only very slowly. The same considerations apply to the bases, except that at 100°C. the uptake of bases of larger molecular size can be assisted by carrier techniques.

Work so far in this field has suggested interesting possibilities if temperatures above 100°C. are considered. It seems likely that at 120–130°C., without resorting to carriers, a very much wider choice of coupling components and bases is available, and this would mean a much wider choice of shade and fastness. It should be added that with the coupling components and bases of small molecular size, which can in any case be applied satisfactorily at 100°C., there does not appear to be any advantage in exceeding this temperature.

OTHER SYNTHETIC AND MAN-MADE FIBRES

Of the other synthetic and man-made fibres which are commonly met with, cellulose acetate can be dismissed on account of its thermoplastic nature and ease of hydrolysis. In the case of nylon and Perlon the problem is not one of dyeability as is the case with Terylene, and the advantages of high-temperature dyeing are similar to those obtained in wool dyeing, i.e. saving in time and increased levelness.

Another class of synthetic fibres where high-temperature dyeing does offer a significant advantage, albeit less striking than with Terylene, is the polyacrylonitrile fibres, e.g. Orlon. Orlon is very similar to Terylene in having a very compact structure and being hydrophobic, i.e. it does not swell appreciably in water, so that the diffusion of dyes within the fibre is slow at normal dyeing temperatures. Again, whilst we do not have such detailed information as is available for Terylene, it is apparent that the dyeing of Orlon materials above 100°C. with disperse or basic dyes offers considerable promise.

Conclusions

The advent of high-temperature dyeing, i.e. dyeing above 100°C., has opened up interesting possibilities for the future in the dyeing of a wide variety of textile materials.

The advantages of high-temperature processing lie in the shorter dyeing times needed and the greater chance of success. In addition, with certain of the newer synthetic fibres, it offers a convenient means of obtaining satisfactory results over a wide range of colours without having recourse to carrier dyeing.

Interesting applications of high-temperature dyeing where worthwhile advantages accrue are the package dyeing of cellulosic yarns and loose stock with vat dyes, the application of direct dyes to viscose rayon materials, in particular filament cakes, the application of acid and chrome dyes to wool yarns, and the dyeing of yarns and fabrics constructed from synthetic materials such as Terylene and Orlon.

Finally, a word of warning: the technique should not be regarded as a panacea for all the ills of dyeing; the technique requires some care in its operation, and in the selection of dyes, if dyed fibres of satisfactory shade, fastness, and physical properties are to be obtained. The process is not of universal application, and in a great number of cases is unlikely to supersede established dyeing methods.

The author wishes to thank his colleagues for their advice and assistance in the preparation of this paper.

DYEHOUSE DEPARTMENT
IMPERIAL CHEMICAL INDUSTRIES LTD.
DYESTUFFS DIVISION
HEXAGON HOUSE
MANCHESTER 9

(*MS. received 4th January 1955*)

References

[1] Royer, Zimmerman, Walter, and Robinson, *Text. Research J.*, **18**, 598 (1948).
[2] AATCC Philadelphia Section, *Amer. Dyestuff Rep.*, **38**, P 9 (1949).
[3] Groeninghe Ververij, *BP* 678,952.
[4] Idem, *BP* 679,687.
[5] Drijvers, *Teintex*, **17**, 294 (1952); *Amer. Dyestuff Rep.*, **41**, 533 (1952).
[6] Cole, *Dyer*, **108**, 925 (1952).
[7] Grand, *American Dyestuff Rep.*, **42**, P 543 (1953).
[8] Walter, *USP* 2,387,200.
[9] Ashpole, McFarlane, and Wilcock, J.S.D.C., **66**, 17 (1950).
[10] Armfield, *ibid.*, **67**, 297 (1951).
[11] Butterworth, *ibid.*, **69**, 362 (1953).
[12] Herrmann, *Melliand Textilber.*, **33**, 1110 (1952).
[13] Society of Dyers and Colourists, *Reports of the Committee on the Dyeing Properties of Direct Cotton Dyes* (reprinted from J.S.D.C., **62**, 280 (1946) and **64**, 145 (1948)).
[14] AATCC Piedmont Section, *Amer. Dyestuff Rep.*, **38**, P 213 (1949).
[15] American Cyanamid Co., *BP* 679,683.
[16] Idem, *BP* 693,251.
[17] Zimmerman, Mecco, Fordemwalt, and Cooke, *Amer. Dyestuff Rep.*, **41**, 597 (1952).
[18] Blackburn and Fox, J.S.D.C., **71**, 80 (Feb. 1955).
[19] Senior, *Textile J. Australia*, **29**, 340 (1954).
[20] Coutie, Lemin, and Sagar, J.S.D.C., **71**, 433 (Aug. 1955).
[21] Iannarone, Clapham, and Thomas, *Amer. Dyestuff Rep.*, **42**, 666 (1953).
[22] Waters, J.S.D.C., **66**, 609 (1950).

Courpleta—The Dyeing and other Properties of Cellulose Triacetate Yarn and Staple

J. BOULTON

Meeting of the Manchester Section held at The Textile Institute, Manchester, on 15th April 1955, Mr. J. W. Reidy in the chair

The manufacture of Courpleta* is briefly described, and a short historical review given of the development of cellulose triacetate fibres. Detailed physical and chemical properties of the fibre are enumerated. In overall behaviour as a textile fibre, Courpleta closely resembles the synthetic fibres. On the other hand, its dyeing properties are more nearly like those of secondary acetate. This spread of properties, with especial emphasis on the ready dyeability of the fibre in contrast to synthetics, and its capacity to provide durable pleating and embossing effects, establish it, in view of its price, as a fibre of great promise. The techniques of dyeing so far developed are discussed in detail, and a description is given of durable pleating effects. Tables of uses and colour fastnesses are provided.

I—Introduction

Cellulose acetic ester was first made in the form of triacetate by Schutzenburger in 1869, and the commercial process for its manufacture was first described by Cross and Bevan in 1894. Essentially, their process, the action of acetic anhydride in the presence of a sulphuric acid catalyst, is still used for the acetylation of cellulose. Triacetate did not succeed as a fibre because of the insolubility of the fully esterified product in any organic solvent capable of use for spinning by solution extrusion followed by rapid solvent evaporation. The Lustron Co. (U.S.A.) attempted its commercial exploitation using the then only available low-boiling solvent, chloroform. The early triacetates were really mixed esters of acetic acid and small quantities of sulphuric acid, employed as catalyst. As such they were unstable to heat, this being a consequence of the formation of sulphuric acid in the fibre when hot and moist. Soon after the turn of the century it was discovered that if fully acetylated cellulose were hydrolysed from an acetyl content of 3 to ca. 2·4 radicals per glucose residue, the product was soluble in acetone, a cheap, safe medium for spinning. The patents of Bayer and Miles (1905) for producing this "secondary" acetate were not used commercially until the First World War, when a cellulose acetate dope was used for aeroplane skins. The Dreyfus brothers produced the first commercial fibres from this acetone-soluble secondary acetate, and Celanese appeared in 1921.

It is thus apparent that the early development of a triacetate was held up by lack of a useful solvent and insufficient knowledge of the rôle of traces of catalyst in leading to instability. The development of aliphatic chlorohydrocarbons established methylene chloride as a useful solvent for "dry spinning", and the demand for stable acetate powders for heat moulding as plastics, together with intensive research on the optimum conditions for fibre production, led to the establishment of stabilising methods.

It will be seen from succeeding sections covering the properties and uses of Courpleta that this fibre is different from secondary acetate in a number of important ways. In some respects and for some uses the two are comparable, and the new fibre will have to make its own way as a competitor with normal acetate rayon. In overall behaviour as a textile fibre cellulose triacetate is more closely comparable with the synthetic fibres—nylon, polyester fibres, and the acrylics. In this respect Courpleta has two important attributes—it is cheap and it is readily dyed. At the same time it is highly robust chemically and can be heat-set and durably pleated. In tensile properties it is generally similar to other cellulosic and cellulose-derived fibres.

This paper is concerned particularly to discuss the dyes, and dyeing methods, best used on Courpleta and the application of heat treatments in finishing. It will be seen that the fibre, in dyed and undyed form, is suited to a wide range of textile end-uses.

II—Manufacture

1. ACETYLATION

The starting material for fibre spinning is solid cellulose triacetate produced by one of two processes. In both methods a cellulose of uniform properties is required, the reactivity of which may be enhanced by pretreatment with acetic acid; cotton linters and specially pure grades of wood pulp are used.

(*a*) *Non-solvent Process*

Activated cellulose is esterified with acetic anhydride in the presence of an inert solvent for the anhydride in which cellulose triacetate itself is insoluble, but which has some swelling action on the acetylated fibre, e.g. benzene, carbon tetrachloride. The acetylation catalyst may be sulphuric or any of a number of other acids—aromatic sulphonic acids, perchloric acid, zinc chloride, etc. After acetylation the fibrous product must be

**Courpleta* is the registered trade-mark used by Courtaulds Ltd. to denote their cellulose triacetate yarns and staple, details of which were published in the press towards the end of 1954. Courtaulds began the commercial development of triacetate yarns and staple in 1950, after several years' research work. Prior to the adoption of this trade-mark, the products were known in the trade under the code letters "JPS". The trade-mark *Courpleta* is applied to cellulose triacetate in the form of bright or matt continuous-filament yarns and staple.

stabilised by the removal of catalytic acid, and this may be done by heating in a non-solvent medium with acetic acid and/or an ester such as ethyl acetate [1].

(*b*) *The Solvent Process*

Acetylation takes place in the presence of a solvent for the cellulose ester, which may be acetic acid itself or methylene chloride. The sulphuric acid catalyst combines with the cellulose to a degree sufficient to give an unstable product; the stabilisation process consists in a brief hydrolysis of the resulting sulphuric ester groups in solution, before precipitation of the cellulose triacetate.

2. SPINNING

The spinning solution is prepared from fibrous or flake triacetate as an approx. 20% solution in methylene chloride, which solvent may contain some alcohol. The dope is filtered and then dry-spun by extrusion and solvent evaporation on the principles used for secondary acetate spinning, with the necessary modifications called for by the substitution of methylene chloride for acetone as spinning solvent.

The extruded fibre is treated immediately with an antistatic lubricant, which can also carry a fugitive dye for identification tinting. In the case of continuous filament, the yarns are separately collected from each tube, the spinnerets on which determine denier and filament number, and then wound on bobbins. Twist is inserted during the passage from spinneret to bobbin.

For staple fibre production, a number of ends are collected into a tow as they leave the spinneret, no twist is inserted, and the tow is cut to the desired staple length. The staple is crimped to an extent which has been determined by performance in subsequent carding and drafting, and by appropriateness for purpose in manufactured yarns and fibres.

III— Fibre Properties

1. MECHANICAL PROPERTIES

(measured on bright continuous filament)

TENACITY	
Dry	1·3 g./denier
Wet	0·9 g./denier
Knot	1·1 g./denier
EXTENSIBILITY	
Dry	25%
Wet	38%
Knot	22%
DENSITY	1·28 g./c.c.

INITIAL TENSILE MODULUS

0·39 g./denier per 1% extension

2. MOISTURE RELATIONS

Standard regain at 65% R.H.	4·5%
Water imbibition	15·0%

3. EFFECTS OF HEAT

Melting point ca. 300°C.

Significant, and technically important, changes take place in cellulose triacetate when it is heated below its melting point. At ca. 180°C. when dry the molecular arrangement changes towards a more ordered state, internal stresses are released, and the increase in fibre crystallinity is marked.

Heat-treated fibre possesses an increased resistance to changes, both chemical and physical, and the technical importance of this increased stability will be dealt with in detail in those parts of this paper concerned with dyeing and thermal finishing processes. In brief, the most important results of heat treatment are—

(i) Introduction of permanent set
(ii) Capacity for durable pleating and figure embossing
(iii) Reduction in the rate of dye adsorption and desorption
(iv) Increase in colour fastness of dyed fibre.

The presence of moisture during heating probably has a plasticising effect, and more durable set may be obtained at lower working temperatures by steam heating than by dry heating.

4. CHEMICAL PROPERTIES

The behaviour of Courpleta towards the major chemical agencies is as follows—

Boiling Water

There is no hydrolysis of the ester and no dulling, as with secondary acetate. The fact that Courpleta cannot be piece-dulled should be kept in mind when considering end-uses. Its high resistance to heat and water means that it can be (*a*) dyed at temperatures above the boil; and (*b*) used in fabric constructions which undergo woollen and worsted treatments, such as scouring, backwashing, blowing, crabbing, and milling.

Alkalis

Dilute alkaline liquors as used in scouring and dyeing have no appreciable effect on the fibre mechanical properties.

In the application of vat dyes to yarns and fabrics containing Courpleta, no loss of strength is caused by baths of the usual alkalinity. At high temperatures, some surface saponification may take place with caustic soda. In cross-dyeing with viscose rayon and cotton, e.g. with vat or azoic dyes, where saponification is to be avoided absolutely, the following conditions do not affect the fibre— 5 g. sodium hydroxide per litre at 60°C., the normal maximum for vat dyeing; and 20 g. sodium hydroxide per litre at 20°C., which covers any conditions required in azoic dyeing.

Acids

Courpleta is resistant to dilute acids, but is degraded by concentrated strong acids. Acid treatments such as are encountered in wool cross-dyeing using sulphuric, acetic, or formic acid, or in diazotisation, have no effect on the fibre. If sulphuric acid is allowed to dry into the fibre, as in the carbonising process, it will cause degradation. Glacial acetic and formic acids dissolve triacetate.

Oxidising Agents

The intrinsic colour of Courpleta is a good white, and bleaching will normally be undertaken only to whiten another fibre with which it may be blended. It is stable to all commercial bleaching procedures using peroxides, chlorite, and hypochlorites. If it should be necessary for a given sample of triacetate itself to be bleached, sodium chlorite is the most effective agent.

Sodium nitrite, under conditions used for diazotisation and vat dye development, does not affect the fibre.

Solvents

The effects of treatment with some of the common organic solvents are as follows—

24 hr. at 25°C.	
Alcohol	Slight swelling
Acetone	Marked swelling
White spirit	No change
Trichloroethylene ...	Slight swelling
Tetrachloroethylene (perchlorethylene) ...	No change
Chloroform	Dissolves
Methylene chloride ...	Dissolves
Acetic acid (glacial) ...	Dissolves
30 min. at 95°C.	
Acetic acid (5%)	Slight delustring
Cellosolve	Slight swelling
m-Cresol	Dissolves
Formic acid (100%) ...	Dissolves

In dry cleaning, white spirit or tetrachloroethylene should be used. Cold trichloroethylene has no serious effect on the fibre itself, but may weaken it when warm, and because of its slight swelling action tends to remove dye from coloured goods.

5. MICROBIOLOGICAL ATTACK

It is known[2] that acetylation of cellulose produces, progressively, increase in resistance to microbiological attack. Using a soil-burial method, cotton, viscose rayon, secondary acetate, and Courpleta triacetate were examined side by side for decay resulting from the attack of micro-organisms found in soil. The cellulosic fabrics were disintegrated in less than three weeks, secondary acetate fabrics lost 80% and more of their strength in five weeks, and in this same time triacetate fabrics lost less than 5% of their strength.

6. PHOTOCHEMICAL RESISTANCE

All conditions of exposure (climate, wavelength) have not yet been explored, but work to date has shown that Courpleta, either bright or dull, is more resistant to the action of light than is secondary acetate.

7. FORMS COMMERCIALLY AVAILABLE

FILAMENT YARN—Production is in the following deniers, all of which are available as bright or dull yarns—

Denier	Filament No.
45	11
55	13
75	18
100	24
140	32
200	45
300	70

Twist 0·5–1·5 t.p.in.

STAPLE FIBRE—Deniers from 3 to 18 are available. The cotton, wool, worsted, and flax systems are covered by the following production characteristics—

Denier	Staple Length
3	1 3/16, 2, 2½, and 4 in.
4½	2, 2½, and 6 in.
6	6 in.

In Table I some properties of Courpleta are compared with those of the other established rayons and synthetic fibres. The properties discussed above are reflected in the technical dyeing and processing behaviour, briefly as follows—

(*a*) Tensile strength compared with nylon and Terylene (ICI) is low; but, lying between those of secondary acetate and viscose rayon, it is high enough to meet the wide range of textile needs already met by the natural fibres and the established rayons.

(*b*) Resistance to wet processing is high: unlike secondary acetate and like the synthetics, triacetate may be treated in boiling liquors and at temperatures higher than 100°C.

(*c*) Relatively low water imbibition makes for quick drying.

(*d*) Its thermal properties allow durable heat setting, pleating, and embossing.

TABLE I

Properties of Staple Fibres

Fibre	Staples* Staple Length (in.)	Dry Tenacity (g./denier)	Dry Extensibility (%)	Dry Knot Strength (g./denier)	Wet Tenacity (g./denier)	Wet Extensibility (%)	Water Imbibition (%)	Melting Point (°C.)	Capable of Durable Heat Setting and Pleating
Acetate rayon	2½	0·97	29	0·9	0·53	40	25	240	No
Courpleta	4	1·2	21	1·1	0·8	29	15	300	Yes
Viscose rayon	2½	2·2	19	1·8	1·2	22	94	—	No
Nylon	4	3·4	63	3·0	2·9	55	12	250	Yes
Terylene	4½	4·2	38	4·0	3·8	36	5	250	Yes
Orlon 42	2	1·9	37	1·7	1·6	33	13	Softens below 150	Yes

* All of 3 denier

(*e*) Cellulose triacetate dyes readily with disperse dyes: its water imbibition is lower than that of secondary acetate and it dyes more slowly. But it dyes much more readily than polyester and acrylic fibres; established dyeing methods are applicable, and relatively high wet fastness is further improved by heat treatments.

(*f*) In staple form, it can be spun on any system; in blends with cellulosic or protein fibres it withstands any dyeing and finishing treatments applicable to the blend fibre.

IV—Dyeing

The early cellulose acetate fibres, which were produced on an experimental scale at the beginning of this century, were almost certainly close to triacetate, and they were described in the literature of the time as almost impossible to dye. This is not surprising, since at that time the number of dyes available was extremely limited, and fundamental knowledge of dyeing processes, which might have provided a lead, was practically non-existent. It is interesting to note, however, that attempts were made to dye these fibres after pretreatment with swelling agents such as phenols or amines, either alone or in aqueous solutions. Such methods are, of course, the precursors of the methods used at present to promote the dyeing of some of our less easily dyed synthetic fibres.

Immediately after the First World War, the commercial production of cellulose acetate fibres was undertaken. Amongst the fibres produced, the American Lustron, described as approximating to a cellulose triacetate[3], appears to have been considerably more easily dyed (with the dyes then known) than the secondary acetates of the same period, such as Celanese and Rhodiaceta, since it could be dyed to comparatively deep colours with basic dyes, whilst they could not[4].

It is likely that the difference was due not to the difference in acetyl content, but to the higher content of acidic sulphate groups, derived from the acetylation catalyst[5], contained in Lustron. Whilst this high content of acid groups was advantageous in promoting dyeing, it also resulted in a lower thermal stability of the fibre. At this stage, and in spite of the difficulties of production and the lower thermal stability, the triacetate fibre stood in an advantageous position in respect of its dyeability and greater resistance to "blinding" and saponification.

However, the advent of the Ionamines[6], closely followed by the disperse dyes, as we now know them, soon restored the balance of dyeability, and as the commercial possibilities of secondary acetate in respect of its cheapness of manufacture, useful cross-dyeing behaviour, and effects to be obtained by delustring and saponification came to be realised, interest in triacetate fibres declined. The Lustron Co. was taken over by the Celanese Corporation of America in 1924.

Since that time developments in the manufacturing processes have made it possible to market products such as Courpleta having an acetyl content very close to the theoretical maximum and containing very few residual acidic groups derived from the catalyst. Courpleta therefore exhibits a very low affinity for basic dyes, and on the other hand has very good heat stability.

Courpleta can be dyed most readily with the disperse dyes. It can also be dyed with selected azoic combinations and with vat dyes of the indigoid and thioindigoid classes[7]. Water-soluble dyes, such as the Solacet (ICI) and certain mono-sulphonated acid dyes which are sometimes used to dye secondary acetate, have practically no affinity for Courpleta.

In general dyeing behaviour, Courpleta can be regarded as intermediate between secondary cellulose acetate and Terylene. For example, at a temperature of 75°C., at which Seraceta may readily be dyed to full colours, the dyeing of Courpleta using selected rapid-dyeing dyes is restricted to pale and medium depths, whereas Terylene at the same temperature is practically undyed in the absence of a carrier[8]. It is quite permissible to dye Courpleta at the boil by reason of its greater stability, and at this temperature full colours can be readily obtained with selected dyes; the depth of colour obtainable on Terylene at the boil without carrier is about the same as that on Courpleta at 75°C. In fact, when dyed at or near the boil, the majority of disperse dyes can be used for pale and medium depths on Courpleta, the only restrictions being those imposed by price or fastness considerations. For economical production of full colours some selection of dyes is necessary, particularly amongst the blues, which as a group do not build up well on Courpleta. In common with most other dyeing systems, it is advantageous when dyeing heavy depths to work in the shortest possible liquor. Choice of dispersing agents is dictated to some extent by the nature of the material being dyed and the equipment used. For example, soap is a natural choice for the pack-dyeing of slubbing or loose stock, or for yarn package dyeing, owing to its superior dispersing action compared with synthetic products, and in spite of its restraining action it has been found satisfactory for most dyes and for all except the heaviest depths. In these cases it may be necessary to use a synthetic, preferably an anionic dispersing agent such as a sulphated fatty alcohol. In piece dyeing, the usual addition of sulphated fatty alcohol generally provides sufficient dispersing action and has less restraining action than soap. When dyeing is done at the boil, the non-ionic dispersing agents are not so satisfactory as the anionic agents, since some precipitation of dye (or perhaps a dye–dispersing agent complex) on the goods may occur and reduce the fastness to rubbing.

Even when dyeing the palest colours it is advantageous to dye at the boil, since, in addition to the more rapid dyeing, greater ease of levelling, and better build-up obtained, the resultant dyeings have better fastness, to most agencies, than those dyed at lower temperatures. Whilst in yarn, slubbing, and loose-stock dyeing machines it is

TABLE II

Fastness of Disperse Dyes on Bright Courpleta*

Dye	Makers	Fastness to—						
		Daylight		Washing	Burnt	Thermal Finishing†		
		Pale	Medium	S.D.C. No. 2 (staining only)	Gas Fumes	Setting (1 min. at 200°c.)	Pleating (20 sec. at 170°c.)	Pleating (10 min. at 15 lb. steam pressure)
Dispersol Fast Yellow A	ICI	4	5	4	5	4–5	5	4
Dispersol Fast Yellow G	ICI	4–5	5	3–4	5	—	3	—
Serisol Fast Yellow GGL	YDC	6+	6+	3–4	5	5	5	5
Serisol Fast Yellow 4RGL	YDC	6+	6+	4	5	5	5	5
Supracet Yellow 4R	LBH	4–5	6+	3–4	4	5	5	5
Cibacet Yellow GN	Ciba	4–5	5	4	5	4	4–5	5
Artisil Direct Yellow RGFL	S	4–5	6	3–4	5	5	5	5
Celanthrene Fast Yellow GL	DuP	6+	6+	1–2	5	3	4–5	3
Latyl Yellow YL	DuP	6+	6+	4	5	4–5	4–5	4
Dispersol Fast Orange B	ICI	4	5	4–5	5	4–5	4–5	5
Dispersol Fast Orange A	ICI	3–4	4	4	5	4	4	5
Serisol Fast Orange RD	YDC	3–4	4	1–2	3	—	—	—
Cibacet Orange 2R	Ciba	4	5	2	2	3–4	3–4	4–5
Artisil Direct Orange RFL	S	4–5	5	4–5	5	4–5	4–5	5
Dispersol Fast Orange Brown R	ICI	3–4	4	2–3	5	3	4	4–5
Cibacet Golden Brown R	Ciba	2–3	2	3	5	3	3–4	4
Duranol Red 2B	ICI	4–5	5–6	1–2	3	3	3–4	2
Duranol Red X3B	ICI	3–4	4–5	1–2	2	5	4–5	5
Serisol Fast Red 2RDX	YDC	2–3	3	3	4	4	4–5	5
Serisol Fast Scarlet BD	YDC	3–4	4	2	4–5	—	3	—
Serisol Fast Red GGL	YDC	5	5–6	3	5	—	—	5
Serisol Rubine 3BD	YDC	3	4	2–3	5	5	5	5
Cibacet Red BBS	Ciba	3–4	3	4	5	—	—	—
Cibacet Scarlet G	Ciba	2–3	4	2–3	1–2	—	—	—
Duranol Violet 2R	ICI	5	5–6	1–2	1–2	—	3–4	—
Duranol Violet RN	ICI	2–3	2–3	2	1	—	5	—
Serisol Violet RGL	YDC	5	5–6	2–3	5	3–4	4	4
Cibacet Violet 5R	Ciba	5	5	3–4	5	4–5	4–5	5
Celanthrene Violet BGF	DuP	5–6	5–6	4	5	5	4–5	5
Interchem Acetate Blue BGLF	IC	5	6	3–4	5	5	5	5
Latyl Blue FL	DuP	5–6	6	4–5	5	5	5	5
Duranol Blue GN	ICI	3–4	4	2–3	2	3	4–5	3–4
Duranol Blue G	ICI	3	3–4	2–3	2–3	—	4–5	—
Duranol Blue 2G	ICI	3–4	4–5	4	2	4–5	4–5	5
Duranol Brilliant Blue CB	ICI	2	2–3	3	1	—	5	—
Cibacet Blue 5G	Ciba	3	4	4–5	1	—	—	—
Cibacet Sapphire Blue 4G	Ciba	3	4	2	2–3	3	4–5	3–4
Cibacet Sapphire Blue G	Ciba	2	3	3	1	—	5	—
Cibacet Blue 2R	Ciba	4–5	4–5	4	3	4	5	5
Serisol Brilliant Blue BG	YDC	3–4	4–5	4	1	—	—	—
Serisol Dark Blue B	YDC	3	3–4	3–4	3	4–5	4–5	4–5
Serisol Fast Blue BRL	YDC	5	5–6	4–5	3	4–5	5	5
Serisol Fast Blue Green B	YDC	3–4	4–5	4	2–3	—	—	—
Serisol Fast Blue BGL	YDC	5–6	5–6	4–5	5	5	5	4–5
Celliton Fast Blue FFG	BASF	4–5	5	4–5	3	4	4–5	5
Artisil Direct Blue GFL	S	3–4	5	4	5	4	4–5	5
Artisil Direct Blue 4GP	S	3–4	5	2	2–3	5	4–5	5

* This table summarises the properties of a range of disperse dyes on bright Courpleta. Whilst many of the fastness values given have been amply proven in use, the table as a whole is put forward as provisional. In some cases the results are based on laboratory tests without the benefit of consultation with the respective dye manufacturers, but we have every confidence in the overall picture presented by this table.

† Fastness to thermal finishing was rated as follows—
5 = Very good
4 = Good
3 = Moderate
2 = Poor

usual to dye at the boil, this does mean that, in piece dyeing, enclosed equipment is necessary, particularly when dyeing on the jigger.

Courpleta may also be dyed with disperse dyes at temperatures above 100°C. where suitable equipment is available. The advantages to be gained by this procedure are improved overall fastness, particularly to dry cleaning with trichloroethylene, owing to the better penetration of dyes into individual fibres; and the possibility of using very slowly diffusing dyes, which may be necessary to obtain certain colours and fastnesses and which do not build up sufficiently well at the boil. Temperatures up to 130°C. may safely be used in such cases.

A method analogous to the Thermosol (DuP) process for dyeing Terylene [9] can be applied to Courpleta. The fabric is padded with an aqueous suspension of a disperse dye, dried, and heated for a short time at 190–210°C.

The wash fastness of Courpleta dyed with disperse dyes at or near the boil is distinctly better than that of secondary acetate dyed to the same depth. With few exceptions, medium-depth dyeings are completely fast to the S.D.C. No. 1 washing test (for cellulosic textiles) and many dyes are also fast to the higher temperature conditions of the S.D.C. No. 2 test, although some slight staining of other fibres in contact with the triacetate may occur. Mild alkaline milling treatments may also be carried out without fear of colour change, although with some dyes there may be slight bleeding of dye into the soap. It should be pointed out here that the presence of a large proportion of Courpleta in a blend with wool will retard milling action appreciably. It is not believed that heavily milled effects will be sought in this type of blend, however, and the reduced rate of milling will give increased dimensional stability in use. (It is expected that durably pleated garments will be washed rather than dry-cleaned.)

Light fastness of the disperse dyes, although slightly lower than on secondary acetate, is adequate for dresswear and most types of outerwear, provided reasonable care is exercised in dye selection, and appears to be similar to that obtained with the polyester fibres such as Terylene [8]. An analysis of recent daylight fastness assessments on 70 dyes showed that in pale colours 30 dyes had light fastness of 4 or more, half of these having a fastness of 5 or more, whilst in medium depths 46 had a light fastness of 4 or more and 28 a fastness of 5 or more. It must be noted that the light fastness of dyes on dull yarn or matt staple is for most dyes similar to that on bright yarn. Of the 70 dyes examined 52 had the same light fastness on both bright and dull yarn. Daylight fastness ratings of disperse dyes are given in Table II. Light fastness assessments obtained by the use of a fading lamp must be regarded with caution. Of these same 70 dyes only 18 exhibited the same fastness in daylight and in the fading lamp. Of the remainder, 49 dyes showed a discrepancy of 1 point or more in pale colours. In medium depths the difference was not quite so great.

As on secondary acetate, certain disperse dyes are susceptible to gas-fading. Those which are safe on secondary acetate appear also to be safe on Courpleta, whilst those which are susceptible to gas-fading on secondary acetate fade rather more quickly on Courpleta when used without a protective agent. This difference has been attributed to the greater uptake of nitrogen oxides by the secondary acetate fibres [10], or possibly by the water contained within the fibre under normal humid conditions [11], the water thus behaving as a protective agent for the dye. In the case of Courpleta, which has a lower regain, the protective action would be less. It is recommended, therefore, that when dyeing Courpleta with susceptible dyes, a protective agent or inhibitor, preferably the durable type, be applied before or during dyeing. Although most commercial inhibitors are satisfactory in providing protection, some have rather low substantivity on Courpleta and may not be economical in use. The more substantive inhibitors should be applied at a concentration 1½–2 times that normally used for acetate rayon.

The swelling action of trichloroethylene on Courpleta has already been mentioned. This solvent, which is used extensively in non-flammable dry-cleaning plants, has a marked stripping action on normally dyed Courpleta. The rate of stripping depends upon the particular dyes used, and upon the extent to which those dyes have penetrated the fibre. Courpleta which has been dyed at a high temperature is therefore considerably more resistant to trichloroethylene stripping than that dyed at a low temperature, as shown by the curves in Fig. 1. The rate of stripping is also reduced by heat-treatment of the fibre following dyeing, when the changes which take place in the fibre make the dye within the fibre less accessible to trichloroethylene. Other dry-cleaning solvents such as perchlorethylene (which is also a non-flammable solvent) and white spirit have no stripping action. The dry-cleaners are, of course, aware of this state of affairs [12]. Trichloroethylene has in the past given trouble in the dry cleaning of textiles other than Courpleta, and there is a move afoot to change over non-flammable plants from trichloroethylene to tetrachloroethylene (perchlorethylene).

Although the field is, as yet, largely unexplored, there are considerable possibilities apparent in the application of azoic combinations to cellulose triacetate, by methods similar to those which have been used over the years for secondary acetate. This method of dyeing is of particular interest for the production of heavy depths, especially black, fast to washing and milling, and is therefore important in relation to loose-stock and slubbing dyeing.

Selected bases and coupling components will give blacks when applied by a process similar to that normally used for acetate rayon, using the sequence— dye with base, diazotise, couple. For example, Dispersol Diazo Black A (ICI) diazotised and coupled with 2:3-hydroxynaphthoic acid gives

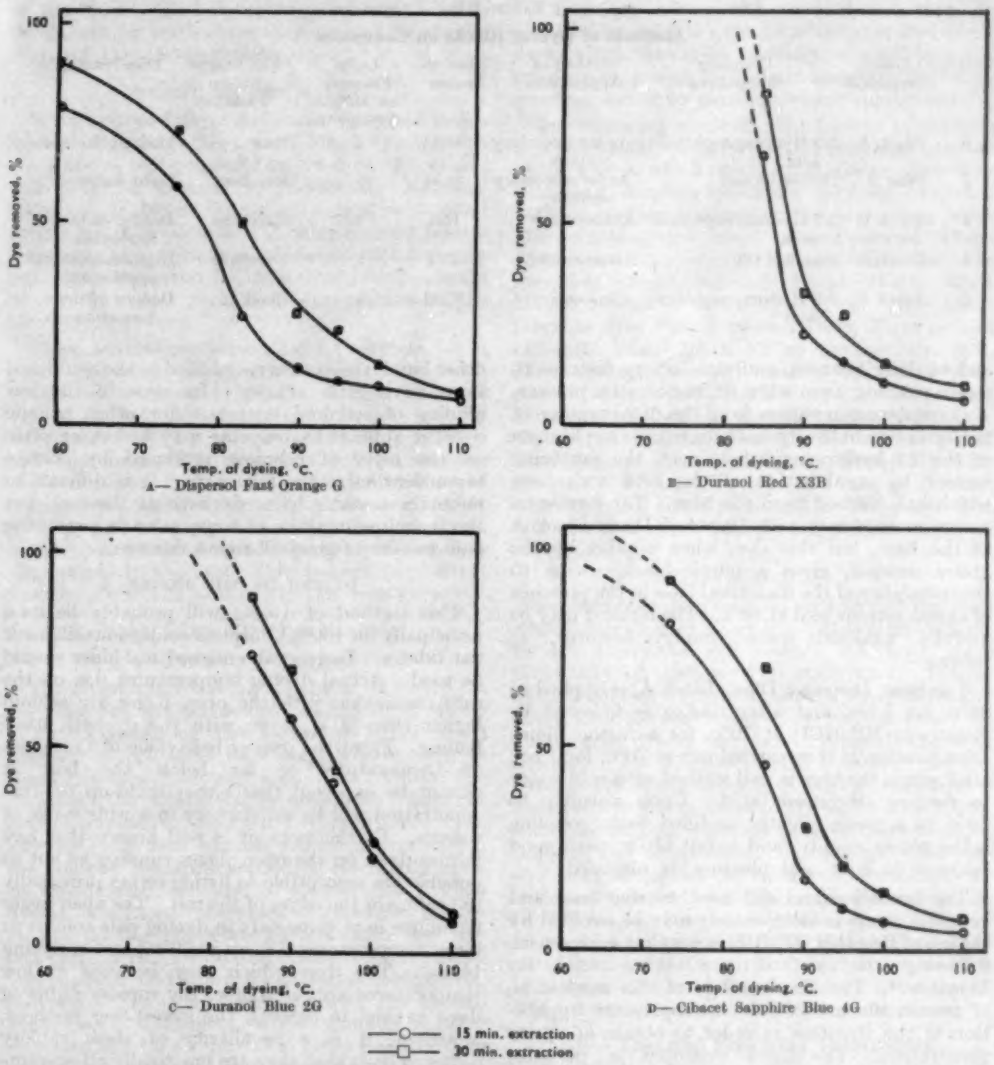


FIG. 1— Effect of Temperature of Dyeing on the Amount of Dye extracted by Trichloroethylene at 20°C.

a black of good light and washing fastness, but not completely fast to steam pleating in the presence of white fibres, owing to some staining of the white. The combination Brentamine Fast Blue B base→phloroglucinol applied by the same method also gives a good black, which, although having lower light fastness than the above, has good fastness to pleating.

Diazotisation of bases on Courpleta takes place rather more slowly than on secondary acetate, and a full hour at 20°C. in a bath containing 1·5 g. sodium nitrite plus 4·5 c.c. hydrochloric acid (32°Tw.) per litre is required for the two above-mentioned bases. The fibre should be well washed with soft water after diazotisation to remove excess nitrite, which would otherwise react with the coupling component (and may result in a brownish black and/or in unsatisfactory wash-fastness).

The alternative method of application, in which base and coupling component are both applied either concurrently or consecutively to the fibre before diazotisation, may also be used. This method, which was introduced many years ago as a means of applying the Naphtol AS series[13] of dyes to acetate rayon, and has recently been used extensively in the dyeing of Terylene, is also suitable for the production of heavy depths, including blacks, on Courpleta. For example, Serisol Diazo Black B (YDC) and 2:3-hydroxy-naphthoic acid applied consecutively at 90–100°C. and 70°C. respectively, and diazotised and coupled simultaneously at 60°C., give a black of good light

TABLE III

Methods of Dyeing Blacks on Courpleta

Method	Diazo Component	Coupling Component	Method of Application	Diazotisation	Light Fastness on Bright Courpleta	Fastness to Steam Pleating	Disadvantages
1	Black A	2:3-Hydroxynaphthoic acid	As for secondary acetate	Cold	4	Poor	Pleating fastness
2	Blue B	Phloroglucinol	As for secondary acetate	Cold	3	Excellent	Light fastness
3	Black B	2:3-Hydroxynaphthoic acid	Consecutive	Hot	6+	Excellent	Danger of tar formation
4	Black B	Brenthol OT	Concurrent	Hot	5	Excellent	High-temperature application
5	Black A	2:3-Hydroxynaphthoic acid	Consecutive	Cold	4	Good	Danger of tar formation

and washing fastness, and satisfactory fastness to steam pleating, even when white fibres are present. This combination suffers from the disadvantage of tar formation in the dyebath during the application of the 2:3-hydroxynaphthoic acid, the tar being formed by combination of the acid with base which has diffused from the fibre. Tar formation occurs more slowly with Dispersol Diazo Black A as the base, but this dye, when applied by the above method, gives a purple–black, owing to the instability of the diazotised base in the presence of excess nitrous acid at 60°C. The method may be usefully modified quite simply, however, as follows—

The base, Dispersol Diazo Black A, is applied at 95°C. for 1 hr., and after rinsing is followed by Brentosyn BB (ICI) at 70°C. for a further hour. Diazotisation is then carried out at 20°C. for 1 hr., after which the fibre is well washed with cold water to remove all nitrous acid. Upon warming to 60°C. in a fresh, slightly acidified bath, coupling takes places readily, and a full black, with good fastness to light and pleating, is obtained.

Tar formation and the need to dye base and coupling component separately may be avoided by the use of Brenthol OT (ICI) as coupling component, following the method now being used for Terylene[14]. The disadvantage of this method is, of course, the need for high-temperature application of the Brenthol in order to obtain adequate penetration. The black produced is, perhaps, not so good as that from Dispersol Diazo Black A and Brentosyn BB, but it has reasonable fastness to light and durable pleating.

The various methods of dyeing blacks, together with fastness properties, are summarised in Table III*. Actual recipes are given in Appendix II.

Azoic combinations, applied by one or other of the above methods, may also be used to produce other colours with satisfactory fastness to light, washing, and pleating.

It is reported[7] that most indigoid and thioindigoid vat dyes may be dyed in pale and medium depths on triacetate fibres at the boil from an alkaline leuco dyebath buffered to a pH lower than 10 in order to avoid undue saponification. On the other hand, the same dyes applied in the acid leuco form have little affinity. In view of the low affinity of cellulose triacetate for other anionic dyes, it appears that dyeing may be taking place on the layer of cellulose produced by surface saponification of the triacetate. It is difficult to maintain a stable leuco dyebath at the boil, but the technique may be of some value in producing pale colours of good all-round fastness.

* Logwood Black has not yet been dyed successfully on Courpleta; whilst direct-dyeing blacks require prolonged application at the boil or at higher temperature and have insufficient fastness to pleating to be of much value.

DYEING ON THE JIGGER

This method of dyeing will probably be used principally for 100% Courpleta continuous-filament flat fabrics. In general, enclosed machines should be used. Actual dyeing temperatures (i.e. on the roll) obtainable with the open jigger are seldom higher than 80°C., even with the dyebath itself boiling. From the dyeing behaviour of Courpleta at temperatures so far below the boil, it cannot be expected that either build-up or fibre penetration will be satisfactory in a wide range of colours. Furthermore, it is well known that any fabrics dyed on the open jigger running as hot as possible are susceptible to listing owing principally to cooling of the edges of the roll. The open jigger technique is of value only in dyeing pale colours at lower temperatures (i.e. up to 60°C.) and in dyeing blacks. The dyes which may be used at low temperatures are, of course, the rapidly diffusing dyes having, in general, the lowest wet fastness. Moreover, it is a peculiarity of these rapidly diffusing dyes that they are practically all susceptible to gas-fading, a pretreatment with inhibitor being required for the best results. A good black, free from listing, with moderately good wet fastness and free from gas-fading troubles, may be dyed on the open jigger by using Dispersol Diazo Black A applied by Method 1 (Appendix II). Whilst not completely fast to durable pleating when white fibres are present, this black has satisfactory fastness on 100% Courpleta fabrics.

Courpleta flat fabrics can be dyed successfully in the enclosed jigger, fabric temperatures near to the boil being obtainable where necessary and difficulties due to listing being eliminated. Under these conditions a much wider range of dyes, many of them free from gas-fading troubles and having better wet fastness, can be applied. No difficulties due to water-marking or undue fabric extension have been experienced when dyeing

at the boil. (End-cloths of secondary acetate should not be used, since they are apt to stretch at the higher temperatures.)

DYEING ON THE WINCH

Warp-knitted fabrics and most Courpleta staple fabrics, including those containing blends with other fibres, will generally be dyed on the winch, and may be treated by essentially standard methods. Fabrics containing a relatively small proportion of triacetate, e.g. warp-knitted fabrics containing Courpleta in combination with Seraceta and/or viscose rayon for cross-dyed effects, should be treated under conditions appropriate to the other fibres present.

Those fabrics containing only Courpleta, or a large proportion blended with Fibro or wool, should be adequately relaxed and given some degree of "set" by treatment in water or soap solution at 90–100°C. in open width before putting into rope form. This treatment tends to improve cover of staple fabrics and prevent formation of rope creases during subsequent processing. After desizing and scouring by normal methods, such fabrics may be dyed with disperse dyes and dyes appropriate to the other fibre present (e.g. direct dyes for Fibro and acid or chrome dyes for wool).

BLENDS AND MIXTURE FABRICS

Courpleta–Seraceta

The difference in dyeing behaviour of Courpleta and Seraceta can be used to obtain dyed effects in fabrics containing these two fibres. When dyed together in the same bath with Solacet dyes or those monosulphonated acid dyes which dye Seraceta, the Courpleta is essentially, but not always completely, reserved. With disperse dyes, however, the dyes exhibit greater individuality, the contrast obtained depending essentially upon the colour used, but also to a lesser extent upon dyeing conditions. Contrast is generally greater at low dyeing temperatures and with low dye concentrations. Yellow and orange dyes in general give the least, and blue and violet dyes the greatest, contrast. This is in agreement with the general behaviour of Courpleta, which exhibits a lower apparent affinity for blue and violet disperse dyes than for disperse dyes of other hues. Desirable two-tone effects can therefore be obtained with disperse dyes. With Courpleta–Seraceta–viscose rayon, the possible range of effects is considerably extended, since the viscose rayon may be dyed independently to any desired colour with direct dyes in the same bath.

Courpleta is not affected by the normal soap–phenol dulling process; hence differential piece-dulled effects may also be obtained.

Courpleta–Fibro

Courpleta is completely reserved by selected direct dyes (e.g. the ICI "CR" range), and may be stained slightly but is not saponified by the normal low-temperature leuco-vat dyeing of viscose rayon. Courpleta reserve effects may therefore readily be obtained over a wide range of colour and fastness.

Two-colour effects and solid shades may be obtained by using a mixture of disperse and direct dyes, which should be carefully selected to avoid cross-staining and to obtain fastness to crease-resisting and/or to pleating (where appropriate).

The following method, used for the production of a solid black on blends with Fibro, is of interest in the way in which two distinct dyeing, diazotisation, and coupling processes may be combined without unduly prolonging the processing time. The combinations used are—Dispersol Diazo Black A→2:3-hydroxynaphthoic acid for the Courpleta and Chlorazol Diazo Black 3B→Direct Developer Z for the Fibro. The blend fabric is dyed with Dispersol Diazo Black A and Chlorazol Diazo Black 3B at or near the boil. After rinsing, the dyed fabric is diazotised cold, rinsed again, and treated cold with Direct Developer Z, which couples under neutral or slightly alkaline conditions on the viscose rayon. The fabric is then treated in a fresh bath with Brentosyn BB, starting cold and raising to 60°C., when coupling takes place on the Courpleta. The fabric is then rinsed and soaped to clear excess of developer.

Courpleta–Wool

Selected acid dyes may be used to dye the wool whilst reserving the Courpleta. It is more difficult to dye Courpleta and reserve the wool, since practically all disperse dyes stain wool. This in itself is seldom important, since a dyed-and-white effect in a 50 : 50 blend may just as easily be obtained by dyeing the wool. When dyeing solid shades with a mixture of acid dyes and disperse dyes, however, the staining of wool by the disperse dyes reduces the fastness of the blend as a whole, since the stained wool has poor fastness to light, rubbing, and perspiration. For some dyes in pale and medium depths, the stain on the wool may be sufficiently cleared by a light soaping after dyeing. In other cases the wool reserve may often be improved by a treatment after dyeing in acidified potassium permanganate, followed by sodium bisulphite. The acid dyes used must, of course, be selected to withstand these clearing treatments.

V—Thermal Finishing Treatments

The effect of dry heat on Courpleta at temperatures up to 150°C. is very slight and of little interest in relation to the finishing of fabrics. Above this temperature the fibre molecules acquire increased mobility, allowing strains to be released and reorientation to take place. The final consequence of this reorientation is that the crystallinity of the fibre, as shown by X-ray diffraction methods, is considerably increased (Fig. 2). Crystallisation takes place quite rapidly at 210–220°C. and more slowly at lower temperatures in the range 180–210°C. The reorientation is accompanied by a fall in the water imbibition of the fibres; for example, on treatment for 30 sec. at 215°C. it falls from its original value of 15 to a more stable value of 12. This in turn results in a considerable reduction in the rate of both absorption and desorption of dyes and an increase in rate of drying. Thus dyed Courpleta heat-treated

at 180–220°C. has better fastness to washing and to trichloroethylene. During the course of the heat-treatment, dye is also distributed more uniformly throughout the fibre, thus reinforcing the effect of the lowered water imbibition upon dye fastness.

Within the temperature region 180–220°C. the fibre is plastic: i.e. it is deformed by small loads, and in this condition may readily be set to desired dimensions, which are substantially retained in subsequent laundering processes. For example, a Courpleta warp-knitted fabric, when machine-washed in a synthetic detergent for 2 hr. at 60°C., had an area shrinkage of 14%. After heat-setting to the same dimensions under a variety of conditions the area shrinkages after washing were as shown in Table IV.

TABLE IV

Area Shrinkage (%) after Heat-treatment

Temperature (°C.)	Time (sec.) 15	30	45
180	4·5	4·4	4·2
190	3·8	3·1	3·2
200	2·9	2·7	2·8
210	3·0	3·0	3·2

With some fabrics, particularly those containing low-twist yarns, where the filaments are almost parallel, heat-setting at 200°C. and above may produce slight adhesion of filaments, resulting in a boardy handle and "finger-marking" (the latter due to breakdown of the filament adhesion under gentle pressure). The adhesion may be broken down by further mechanical working of the fabric, but in any case such fabrics normally have adequate dimensional stability when set at a lower temperature, e.g. 185°C. Treatment at temperatures greatly in excess of 220°C. is undesirable, since no useful improvement in setting is obtained and bursting strength may be reduced.

The heat-setting process also confers better resistance to creasing and in many cases a softer and slightly fuller handle and better drape.

When greater loads are applied, deformation of the fabric may take place at lower temperatures in the region of 160–175°C., the deformation produced again being durable in laundering. Durable-pleating techniques, using machines such as the Rabofsky Rabo 54 continuous pleating machine, fall into this category. Pleats are formed in the fabric, in a predetermined sequence, by knife blades, and the pleated fabric is then passed slowly between a woollen blanket and a heated metal roller, which for Courpleta should be at 165–175°C. In the case of thin fabrics the pleats formed are substantially retained during subsequent washing processes, but thicker fabrics may not be heated through sufficiently, and may require a further heat-setting process to improve the durability of the pleat. If an attempt is made to improve durability by increasing the pleating temperature above 175°C., it is found, particularly with 100% Courpleta fabrics, that the combined effect of heat and pressure produces glazing of the fabric. With blends the pleating temperature may, in some cases, be as high as 185°C. without ill effect.

Durable pleating may also be carried out at lower temperatures using steam at pressures of 10–20 lb./sq. in. Steam appears to act, therefore, as a lubricant or plasticiser, promoting molecular movement. This method of pleating is particularly appropriate to the technique known as "sunray" pleating, in which cut lengths of fabric are folded in manilla formers and subjected to steam under pressure. In addition to the actual pleats produced, the whole of the fabric is set, and garments made in this way, such as ladies' skirts, may be washed at quite high temperatures without appreciable change in appearance or dimensions.

The combined influence of heat and pressure may also be used to produce durable embossed effects, temperatures in the range 170–190°C. and moderate pressures being satisfactory. Durability of the embossing and resistance to creasing during laundering are improved by a post-setting treatment, e.g. by pressure steaming.

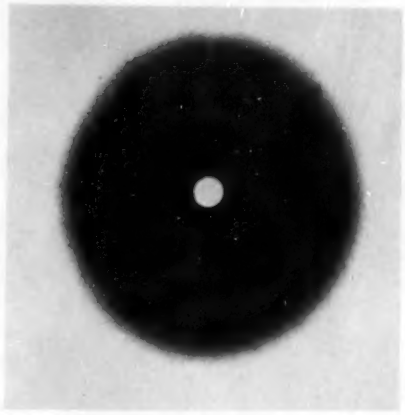
For fabrics containing blends of Courpleta with other, non-setting fibres, the degree of pleat retention or "set" obtained increases continuously with increase in Courpleta content, and the amount of Courpleta required in the blend to obtain satisfactory results depends upon the other fibre present. Thus fabrics of Courpleta–wool require a Courpleta content of 50–60%, whilst a higher proportion, viz. 65–70%, is desirable in Courpleta–Fibro blends.

An important fastness requirement for dyes used on Courpleta may be that they shall withstand the processes of heat-setting and pleating without change. The extent of permissible change is not easy to define, but ideally there should be no change in colour of the material itself, no staining of adjacent fibres, and no staining of the pleating paper. This is rather a lot to ask of any disperse dye, but some dyes, usually the slowly diffusing ones of higher molecular weight, are found to withstand these processes. Many azoic combinations when coupled on the fibre have quite large molecules compared with disperse dyes and also withstand pleating. Care must be taken, however, in the application of such combinations to Courpleta, that no side-reactions resulting in easily diffusible products of low molecular weight take place, otherwise the fastness to pleating and heat-setting processes is considerably reduced.

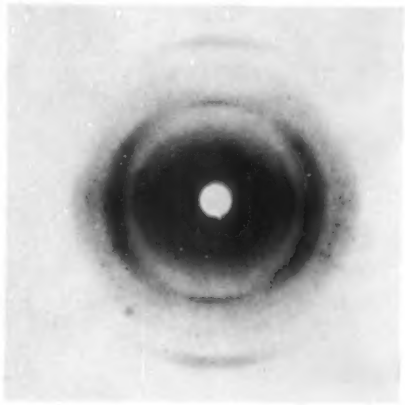
Information on fastness of individual disperse dyes is given in Table II.

VI— Resistance to Creasing

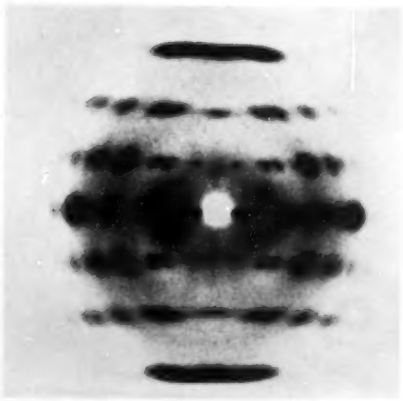
The crease-resistance of all-Courpleta fabrics is good, and is further enhanced by thermal finishing processes. Incorporation of Courpleta, in any proportion, in blends with wool produces fabrics in which resistance to creasing is fully maintained. In the case of blends with Fibro, crease-resistance depends on the proportion of the triacetate fibre; for such uses as skirting and slacks, manufacturers have applied a crease-resist finish to these blends: it should be noted that with a minimum triacetate content of two parts of Courpleta to one part of



(a) Standard Yarn



(b) Yarn heat-treated at 220°C.



(c) Yarn hot-stretched

FIG. 2— Effect of Heat-treatments on the X-Ray Diffraction Pattern of Courpleta

(The photographs were taken using yarn bundles on a 3-cm. flat-film camera with Copper K_α radiation, and fibre axes vertical)

Printofix PD & PF
SANDOZ
RESIN BINDERS FOR PRINTING
Opaque white and pigments;
gold and aluminium metallic powders
Discharge printing on acetate silk
Whites on nylon
Flock printing
Particulars and recipes
on request
Sandoz
PRODUCTS LTD
BRADFORD

face centre

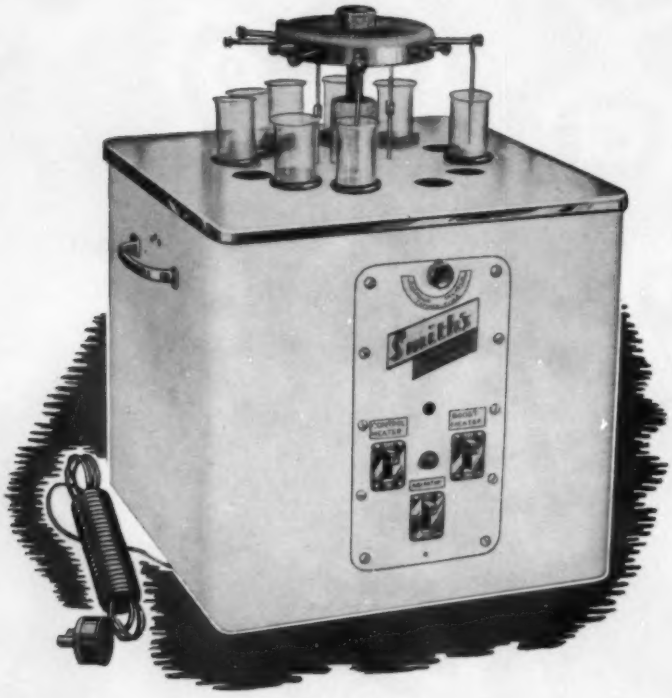
Quick, accurate tests speed your turnover

This new

LABORATORY DYE TESTING MACHINE

Patent applied for

is the answer

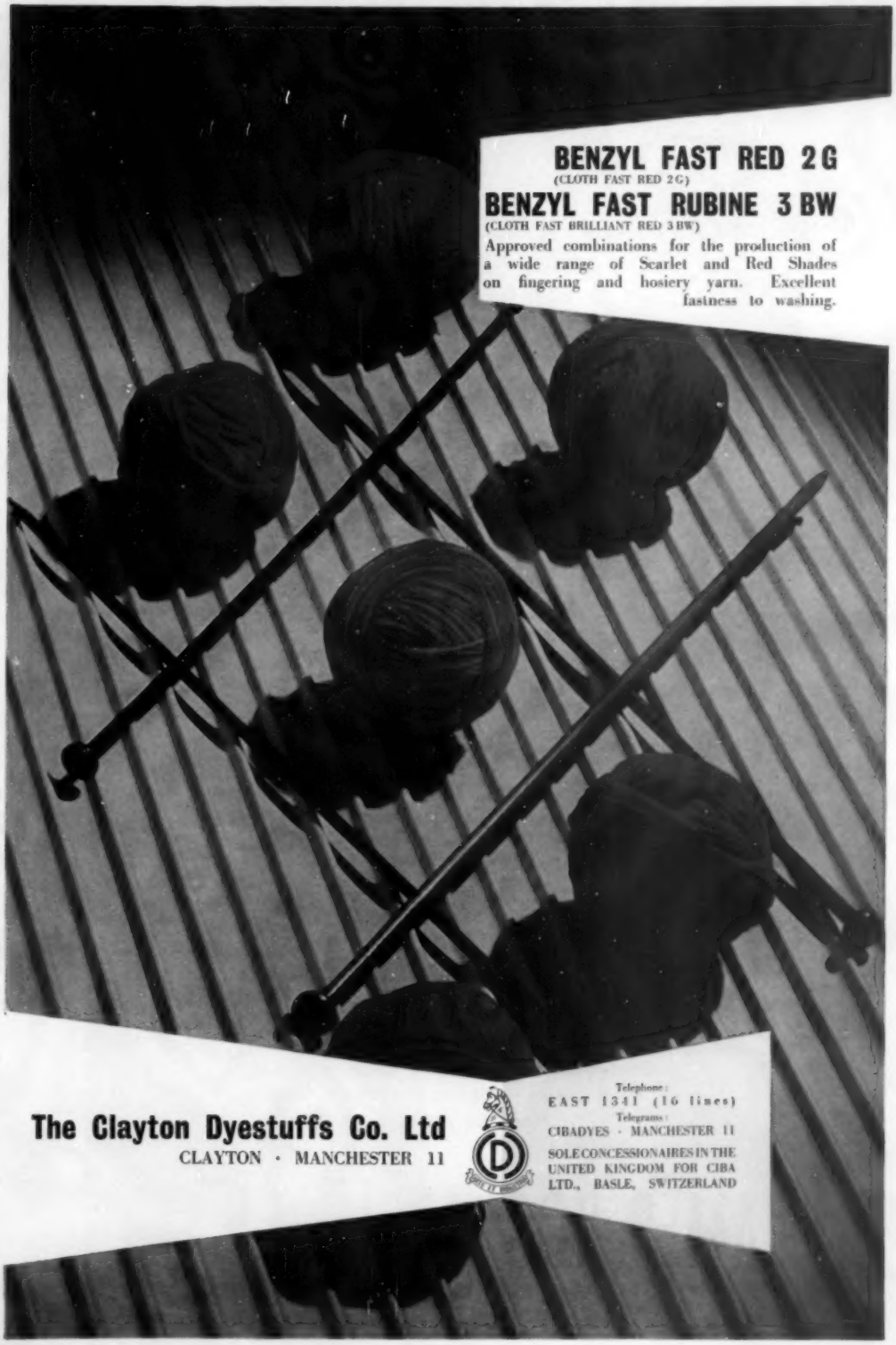


Portable, thermostatically controlled bench model of stainless steel and stove enamel finish for cleanliness. Connects to any 13 or 15 amp socket. Samples may be dyed simply and accurately with a choice of sample holders and vessels to suit your particular requirements

We make a variety of Laboratory machines. Please write to us

F SMITH & CO (WHITWORTH) LTD

WHITWORTH ROCHDALE *Telephone* Whitworth 52233 *Telegrams* AUTOJIG WHITWORTH


BENZYL FAST RED 2G
(CLOTH FAST RED 2G)
BENZYL FAST RUBINE 3BW
(CLOTH FAST BRILLIANT RED 3BW)
Approved combinations for the production of a wide range of Scarlet and Red Shades on fingering and hosiery yarn. Excellent fastness to washing.
The Clayton Dyestuffs Co. Ltd
CLAYTON · MANCHESTER 11
Telephone:
EAST 1341 (16 lines)
Telegrams:
CIBADYES · MANCHESTER 11
SOLE CONCESSIONAIRES IN THE UNITED KINGDOM FOR CIBA LTD., BASLE, SWITZERLAND

Sulphol colours

Cotton and Rayon Dress Goods Cloths for Rubber Proofing

Umbrella Cloths

Nurses Uniforms

also for

Sewing Cotton
Handkerchiefs
Cotton and Union Shirtings
Linings

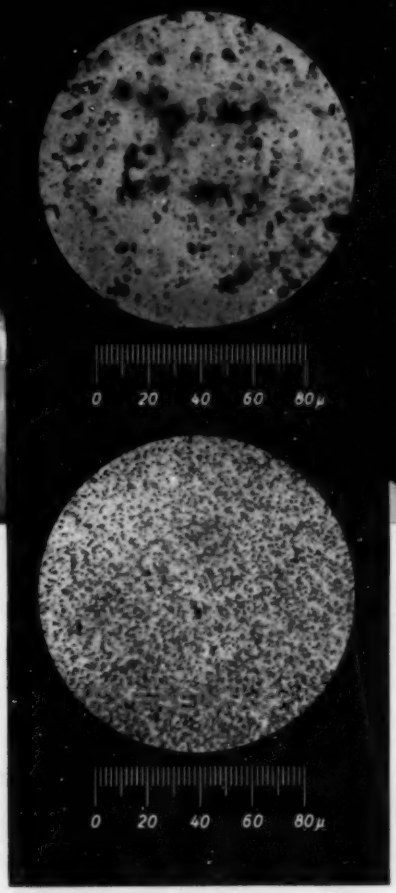
Webbing Belts
Canvas Containers
Haversacks
Wagon and Other Covers

Gabardines Mocquette Backings Linen
Spun Rayon Cloths for Plastic Coating
Fibro for mixed yarns

JAMES ROBINSON & CO LTD

HILLHOUSE LANE HUDDERSFIELD ENGLAND

Telephone 334-335 *Telegrams* ROBINSON HUDDERSFIELD



The high-speed processing of goods in modern continuous dyeing plant calls for vat dyestuffs of exceptionally small and uniform particle size. Cibanone Micro Disperse brands developed from the fastest anthraquinonoid dyes are unexcelled in meeting these requirements.

The photomicrographs from the research laboratories of CIBA Limited, Basle, show an ordinary vat dye alongside a Cibanone Micro Disperse brand.

CIBA Limited, Basle, Switzerland.

The Clayton Dyestuffs Co., Ltd., Manchester, 11.
Sole Concessionaires in the United Kingdom.

C I B A

BRENTAMINE
FAST COLOUR SALTS AND BASES
in combination with suitable Brenthols
★ Extensive fast range
★ For strong, bright azoic shades, especially in 'well covered' patterns
★ Invaluable in a wide variety of direct, resist and discharge printing styles
★ Widely used in specialised prints for the West African Market
Comprehensive technical service freely available
IMPERIAL CHEMICAL INDUSTRIES LTD.
LONDON, S.W.1
ICI

Inns and the Textile Trade

No. 3 *'The Woolpack Inn,' Norwich*

Once it was a ramshackle house of flint and oak edging onto a cobbled street. From its windows, travellers would have seen macabre processions of townsfolk carrying their dead to the reputed plague-pit opposite. Or watched cattle herded through the narrows to Norwich market nearby and pack horses swaying under bales of wool. Men gathered at the inn mainly on business. For the Woolpack was the market house.

And now? The inn is a large Georgian-style building standing back from a modern highway. The market is still there—one of the largest in the country. Norwich continues to prosper from wool-trading. And the "Woolpack" is still the market house.

Its unique inn sign of beaten copper, a great bale of wool, tacitly recalls the days of the little pack horses. Inside, several large wall plaques, fashioned of plaster and painted with infinite care, picture the story of wool and the trade: the sheep, the shearing, spinning and weaving, the Flemings, the sailing ships that exported the cloth.

There is no plaque to depict the chemical processing of wool. But chemical research keeps the ancient trade in line with modern times—important work—carried on by companies such as Brotherton.

Brotherton

One of the world's largest manufacturers of hydrosulphites, liquid sulphur dioxide and hexamine. Makers of an extensive range of Metachrome dyes for dyeing wool in all its forms.

Brotherton & Co., Ltd., City Chambers, Leeds, 1
Telephone; Leeds 2-9321
Telegrams; 'Brotherton Leeds'

Perfect Cones and Cheeses for Dyeing and Bleaching

STUBBS

HIGH SPEED

CONE & CHEESE WINDER

Simple and economical machine, silent in operation.
Auxiliary motion prevents figuring-up and hard edges on the package.
Balanced Winding Head.
Movable yarn plate ensures perfectly built Cones or Cheeses.
Diameter Stop motion.
Winding speed up to 1,000 yards per minute.

The above machine can be seen in operation at our Mill Street Works, and samples of yarn can be wound.

MAKERS OF MACHINERY FOR WINDING, DOUBLING, CLEARING, GASSING, REELING, PREPARING, POLISHING AND BUNDLING ALL CLASSES OF YARNS AND THREADS

JOSEPH STUBBS LTD, MILL STREET WORKS, ANCOATS, MANCHESTER 4

Telephone Collyhurst 1721 (3 lines) *Telegrams* WINDING MANCHESTER

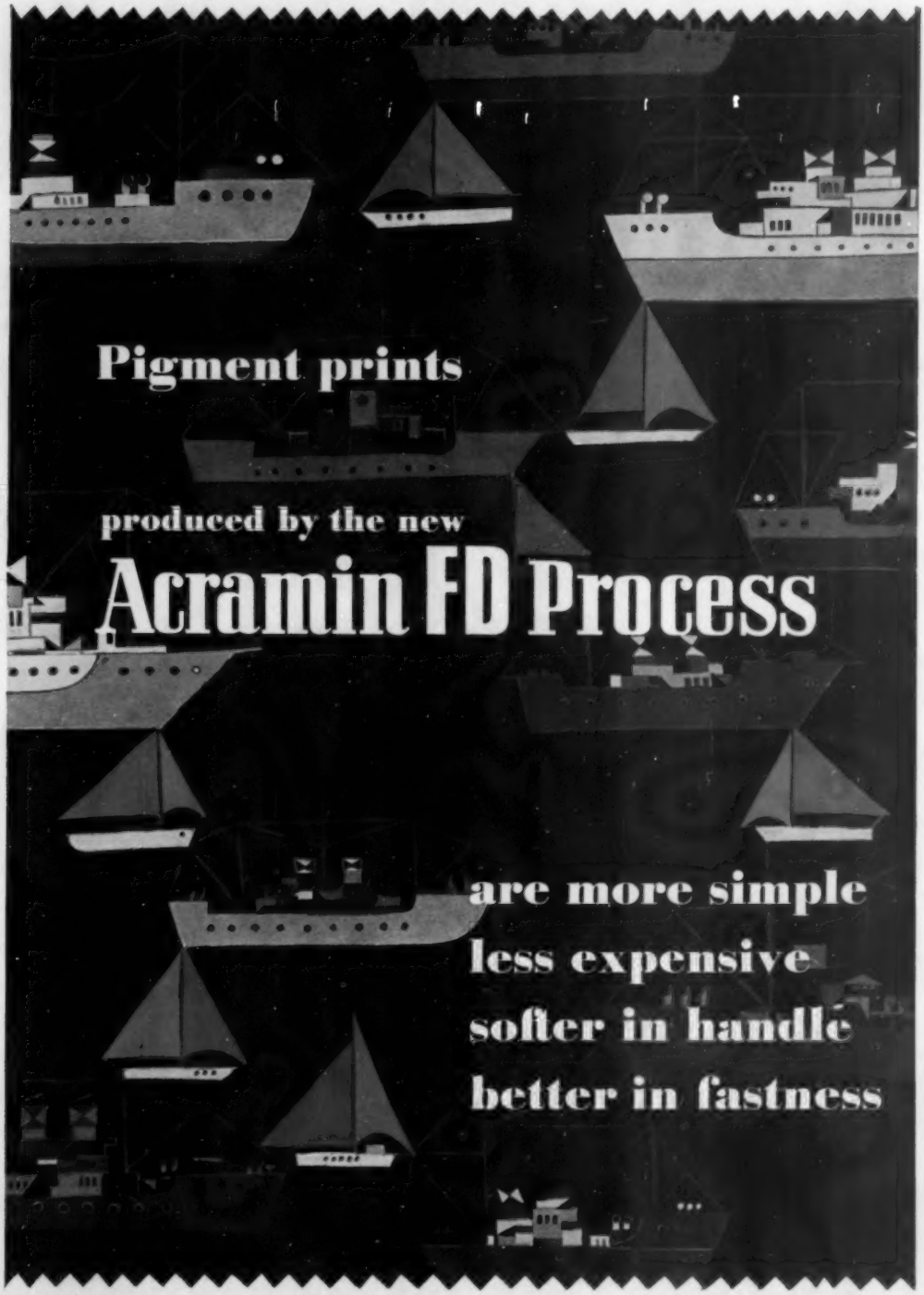
SERISOL BRILLIANT RED X3B

PRINTING PASTE

For direct prints on Acetate, Nylon, Terylene

THE YORKSHIRE DYEWARE & CHEMICAL COMPANY LTD., YDC LEEDS.

For Full details
concerning
HYDROGEN PEROXIDE
dial "A"
for
THE MANUFACTURERS
ALCOCK
'PHONE: LUTON 4900 (3 lines)
(PEROXIDE) LTD
LUTON
BEDS.
GRAMS: 'PEROXIDE' LUTON
AGENTS:-
THOMAS HUNTER & SONS
35-37 BOYNE SQUARE
BELFAST, N. IRELAND
'PHONE: BELFAST 2008
GRAMS: CHEMICALS, BELFAST
F. BRAYSHAW & SON
JUNCTION MILLS
LAISTERDYKE, BRADFORD
PHONE: BRADFORD 65033
GRAMS: OKYGEN, BRADFORD
An Electrolytic product of unsurpassed quality
24 hour service (own Transport) backed by
Depot stocks for more urgent requirements
Free Technical and Laboratory Service



BAYER LEVERKUSEN GERMANY

Distributors in Great Britain: Industrial Dyestuffs Limited
94, Market Street, Manchester, 1 — 38-40, Sunbridge Road, Bradford
27, Oswald Street, Glasgow, C. 1 — Dunster House, Mincing Lane, London, E. C. 3

BAYER

Gay!

a splash of colour...
a subtle lustre...
sea, sun and
soap-sud free

Beetle Textile Resins play an important part in the production of the infinite variety of 'Everglaze' fabrics, glossy and matt, flat and textured. Economical, practical and right in the fashion, these effects can be produced by the 'Everglaze' finisher in a wide variety of printed and dyed materials.

GENERAL APPLICATIONS

Beetle Resins enter into an ever-widening variety of fashion finishes, and combined effects can be obtained by using the appropriate resins together in a single application:

- Crease Resistant Finishes
- Fullness of Handle
- Shrink Resistant Finishes
- Durable Glazed and Embossed effects
- Durable Stiffening of nylon
- Durable Water Repellency on rayon, cotton and mixtures

BEETLE *Fashion* FINISHES

B·I·P Chemicals Ltd (B·I·P) Oldbury · Birmingham
'phone: Broadwell 2061

Manchester Office: 79 Mosley Street, Manchester 2 'phone: Central 9014

Fibro, treatment for crease-resistance is quite unnecessary. Such a blend provides a satisfactorily crease-resistant fabric.

VII—End-uses

These are early days. Courpleta as a commercial textile fibre is in its infancy, but its future use in a comprehensive range of textile applications would seem to be assured. It is clearly advantageous to have a relatively cheap hydrophobic fibre which can be readily dyed, which can be thermally set, pleated, or embossed and retain its modified form in wear and wash, which is crease-resisting in its own right, and which can contribute these characteristics when used in blends with other fibres. End-uses so far explored cover a wide field, and already Courpleta is seen as a universal rather than a specialised textile raw material.

As a simple replacement for continuous-filament secondary acetate, cellulose triacetate can contribute a crisper and fuller handle, better resistance to creasing, better behaviour to ironing in taffetas and lining materials, and dimensional stability in warp-knit fabrics; in conjunction with continuous-filament secondary acetate and viscose rayon a wide range of three- or four-colour piece-dyed effects in both woven and knitted fabrics are obtainable. When used in blends with wool, it does not down-grade the fabric, and provides a ready means of obtaining more durable pleating and better resistance to shrinkage during washing. A blend of Courpleta and Fibro which can be durably pleated has also better dimensional stability and resistance to creasing than an otherwise similar all-Fibro fabric. It should not require crease-resist finishing.

TABLE V

End-use	Fibre Type*	Fastness demanded Light	Washing†	Other Fastness‡
Afternoon frocks ...	F S	3	1	—
Lingerie	F	2	1	—
Blouses	F S	3	1	—
Washable dresses ...	F S	4–5	1	—
Skirts	S	5	1	D
Slacks	S	5	1	D
Tailored frocks ...	S	4–5	1	D
Two-piece suits ...	S	5	1	D
Evening gowns ...	F	–	–	P D G
Coat linings ...	F	–	–	P D G
Men's trousers ...	S	5	1	D
Tropical suitings ...	S	5	1	P D
Sports shirts ...	F S	5	2	D G

* F = Filament yarn
S = Staple, alone and in blends

† Should have a rating of 4–5 or 5 in the appropriate (No. 1 or No. 2) S.D.C. washing test for cellulosic textiles

‡ D = Dry cleaning
G = Gas-fume fading
P = Perspiration

In exploring end-uses for Courpleta it is obviously necessary to consider the colour fastness required in particular types of garment, household goods, etc., in addition to the fastness to thermal finishing processes already discussed. A useful guide to general textile colour fastness demands has been given by Tattersfield[15]. Using the symbols and criteria employed in his fastness schedule, the work done to date on Courpleta has established the list in Table V of recommended end-uses, the fastness requirements demanded for which are attainable on Courpleta and its appropriate blends.

The products given in Table V were all illustrated by garments and fabrics on show at this lecture. It is seen that this fibre, new to the textile industry at large, has gone a long way in a very short time. It will undoubtedly go further. Only the time required for the necessary development work to be done stands between this list of established uses and one which will be much longer. In Courpleta the industry has another fibre with which to expand the range of inexpensive, sound textiles, and the designer and the dyer have another medium with which to work. And, in this day and age, the dyer will appreciate it as a medium on which he can, in fact, exercise his art. Courpleta can be dyed by traditional means. In addition, the techniques of heat setting, durable pleating, and high-temperature pressure dyeing are applicable.

* * *

In reporting some of the work done on Courpleta, the author has drawn widely on the efforts of many colleagues in the Research Department, the Coventry Dyeing Department, and the Use–Development Organisation of Courtaulds Ltd. His thanks are due to the Directors for permission to give the paper. He is, further, indebted to senior people on the staffs of dye manufacturers, in particular Imperial Chemical Industries Ltd., The Clayton Dyestuffs Co. Ltd., and The Yorkshire Dyeware & Chemical Co. Ltd., for valuable discussions.

Appendix I

SELECTION OF DYES

Courpleta can be dyed economically to pale and medium depths with the majority of the available disperse dyes from a bath containing soap or other commercially available dispersing agent. The soap dyebath usually gives more restraining action than other agents.

For heavy depths, the following dyes, having particularly good building-up properties, are recommended—

Dispersol Fast Yellow G (ICI)
Serisol Fast Yellow N (YDC)
Dispersol Fast Orange G (ICI)
Artisil Direct Orange RFL (S)
Dispersol Fast Orange A (ICI)
Dispersol Fast Orange Brown R (ICI)
Serisol Fast Orange GD (YDC)
Duranol Red 2B (ICI)
Serisol Rubine 3BD (YDC)
Serisol Dark Blue B (YDC)
Duranol Violet 2R (ICI)
Duranol Brilliant Violet B (ICI)
Artisil Direct Blue 4GP (S)
Duranol Blue G (ICI)
Cibacet Sapphire Blue 4G (Ciba)

Dyes having a fastness of 3 or less to burnt gas fumes should be protected by an inhibitor, e.g. 2% Duranol Inhibitor GF (ICI) or 3% Serisol Inhibitor (Protac 30) (YDC) applied before or during dyeing.

Appendix II

EXAMPLES OF METHODS FOR PRODUCING BLACKS

Method 1

(e.g. on Courpleta poult fabric dyed on an open jigger)

SCOURING—As for acetate rayon.

DYEING—Set the dyebath with 6% Dispersol Diazo Black A 300 and 1 g. Lissapol C powder (ICI) per litre. Enter the fabric cold, raise the temperature to 90–95°C., and continue dyeing at that temperature for 3 hr. Rinse.

DIAZOTISATION—Treat the dyed fabric for 1 hr. at 20°C. in a bath containing—

1·5 g. Sodium nitrite
4·5 c.c. Hydrochloric acid (32°Tw.) } per litre

(These quantities should be increased when working in liquor ratios of less than 10 : 1.) Wash off thoroughly.

DEVELOPMENT—Set the bath with 5% 2:3-hydroxynaphthoic acid and bring the pH to 3 with hydrochloric acid. Enter the fabric cold, raise the temperature to 90–95°C. in 30 min., and continue developing at that temperature for 45 min.

SOAPING—Soap the fabric in 0·25% soap solution for 30–60 min. at 90–95°C., wash off in cold water, and finish as desired.

Method 2

(e.g. on Courpleta lining fabric on enclosed jigger)

SCOURING—As for acetate rayon.

DYEING—Set the dyebath with 4% Brentamine Fast Blue B base and 1 g. Lissapol C powder per litre. Enter the fabric cold, raise the temperature slowly to 95°C., and continue dyeing at that temperature for a further hour. Rinse.

DIAZOTISATION—Treat the dyed fabric for 1 hr. at 20°C. in a bath containing—

1·5 g. Sodium nitrite
4·5 c.c. Hydrochloric acid (32°Tw.) } per litre

Wash off well in cold water. (These quantities should be increased when working in liquor ratios of less than 10 : 1.)

DEVELOPMENT—Set the bath with 1% phloroglucinol. Enter the fabric cold, raise the temperature to 60°C. in 30 min., and continue development at that temperature for 30 min.

SOAPING—Soap the fabric for 30 min. at 90°C. in 0·25% soap solution, wash off in cold water, and finish as desired.

Method 3

(e.g. on Courpleta slubbing in Obermaier machine)

SCOURING—Scour for 15 min. at 60°C. in a bath containing 1 g. Lissapol C powder per litre.

DYEING—Set the dyebath with 6% Dispersol Diazo Black B 300 in Lissapol C powder solution (2 g./litre) at 60°C. Allow the liquor to circulate at 60°C. for 10 min., warm to 95–98°C. in 30 min., and continue dyeing at that temperature for a further hour. Wash off in cold water. Prepare a fresh bath containing 4% 2:3-hydroxynaphthoic acid in Lissapol C (2 g./litre) and allow to circulate for one hour at 65–70°C. Drop the bath and wash in 5 g. soda ash per litre for 10 min. at 25–30°C., then wash off thoroughly in water at 40°C.

DIAZOTISATION AND DEVELOPMENT—Set the bath with—

2 g. Sodium nitrite
4 c.c. Hydrochloric acid (32°Tw.) } per litre

at 20°C. Allow the liquor to circulate for 15 min. at 20°C., raise the temperature to 60°C. in 20 min., and continue development at that temperature for a further 30 min. Drop the bath and rinse in cold water.

"REDUCTION-CLEARING"—Prepare a fresh bath containing—

5 c.c. Caustic soda (70°Tw.)
2 g. Sodium hydrosulphite
2 g. Lissolamine A 50% paste (ICI) } per litre

at 20°C. Raise the temperature to 60°C. in 15 min. and continue clearing at 60°C. for 15 min. Drop the bath and rinse in cold water.

"SOAPING"—Soap for 15 min. at 80°C. in a bath containing—

2 g. Soda ash
1 g. Lissapol C powder } per litre

Wash off well in cold water and finish as required.

Method 4

(e.g. on Courpleta staple in Longclose pressure dyeing machine)

SCOURING—Scour for 15 min. at 60°C. in a bath containing Lissapol C powder (1 g./litre).

DYEING—Set the dyebath with 6% Dispersol Diazo Black B 300 and 4% Brenthol OT in Lissapol C powder (2 g./litre). The dye and the Brenthol OT* should be dispersed and homogenised before they are added to the dyebath. Allow the liquor to circulate at 60°C. for 10 min., raise the temperature to 120°C. in 40 min., and continue dyeing at that temperature for 30 min. Drop the bath and wash in cold water containing 2·5 c.c. caustic soda (70°Tw.) per litre. Wash off thoroughly in cold water.

DIAZOTISATION AND DEVELOPMENT—Treat for 30 min. at 60°C. in a bath containing—

2 g. Sodium nitrite
4 c.c. Hydrochloric acid (32°Tw.) } per litre

"REDUCTION-CLEARING" AND "SOAPING"—As for Method 3.

Method 5

(e.g. on 100% dull Courpleta warp-knit fabric† on an enclosed winch)

SCOURING—As for acetate rayon.

DYEING—Set the dyebath with 6% Dispersol Diazo Black A 300 in Lissapol C powder (1 g./litre). Commence dyeing cold, raise the temperature to 90°C. in 30 min., and continue dyeing at that temperature for a further hour. Drop the bath and rinse.

Treat the dyed fabric in a fresh bath containing 6% 2:3-hydroxynaphthoic acid in Lissapol C powder (1 g. per litre) for 1 hr. at 65–70°C. At this stage, if tarring has occurred, a cold wash in water containing 5 g. soda ash per litre may be given, followed by a thorough wash in water at 40°C. to remove alkalinity.

DIAZOTISATION—Treat the fabric for 1 hr. at 20°C. in a bath containing—

2·5 g. Sodium nitrite
7·5 c.c. Hydrochloric acid (32°Tw.) } per litre

Drop the bath, rinse the fabric in water, and treat for 30 min. at 60°C. in water slightly acidified with hydrochloric acid.

SOAPING—Soap for 20 min. at 90°C. in 0·25% soap solution. Wash in cold water and finish as required.

RESEARCH LABORATORY
COURTAULDS LTD.
DROYLSDEN
MANCHESTER

(*MS. received 17th May 1955*)

* Brenthol OT is best dispersed by first dissolving it in caustic soda, and then reprecipitating it in a dilute solution of phosphoric acid and Lissapol C as recommended by ICI.

† Courpleta fabrics of this type should be adequately relaxed and given some degree of set by treating in water at 90–100°C. in open width before putting into rope form.

References

[1] Sindl, *BP* 411,260.
[2] Thaysen, *Ann. Appl. Biol.*, **26**, 750 (1939).
[3] Mullin, *Acetate Silk and its Dyes* (New York: D. Van Nostrand Co. Inc. 1927), p. 45.
[4] Mullin, *op. cit.*, p. 137.
[5] Caille, *Chim. et Ind.*, **15**, 61 T and 189 (1926).
[6] Green and Saunders, *J.S.D.C.*, **34**, 10 (1923).
[7] ICI, *Technical Information*, Dyehouse No. 243 (1955).
[8] ICI, *The Dyeing of Terylene Polyester Fibre* (2nd edition 1954).
[9] DuP, *Tech. Bull.*, **5**, 82 (1949).
[10] Salvin, Paist, and Myles, *Amer. Dyestuff Rep.*, **41**, P 297 (1952).
[11] Myles and Reiss, *J. Polymer Sci.*, **25**, 243 (1955).
[12] Davies and Rhodes, *J.S.D.C.*, **71**, 143 (March 1955).
[13] BrC, *BP* 310,379; Wallwork, *J.S.D.C.*, **51**, 415 (1935).
[14] ICI, *Technical Information*, Dyehouse No. 237 (1955.)
[15] Tattersfield, *J. Textile Inst.*, **40**, P 653 (1949).

Discussion

Mr. C. P. ATKINSON: What is the maximum temperature envisaged for the dyeing of Courpleta?

Mr. BOULTON: We have seldom found it necessary to dye Courpleta at temperatures over the boil. However, when such conditions are necessary, as in the case of very slowly diffusing dyes required to achieve special fastness, the temperatures that would appear necessary are in the range 120–130°C.

Mr. B. KRAMRISCH: When loose stock, sliver, or yarns of pure or blended Courpleta are dyed at high temperature, is there any possibility of degradation if the dyebath is not neutral?

Mr. BOULTON: Interest here would be in azoic and possibly acid dyeing, and as only mildly alkaline and acid treatments are involved there is no degradation of the fibre. We have not yet made a full investigation of the effect of pH at temperatures over 100°C.

Mr. H. R. HADFIELD: Is soap recommended as dispersing agent at high temperature?

Mr. BOULTON: The restraining action and the dispersing action of soap, which are of value in assisting level dyeing with the more rapidly dyeing dyes at lower temperatures, offer little advantage in these special cases: we find a synthetic agent, preferably a sulphated fatty alcohol, to be satisfactory.

Mr. S. BURGESS: There is at present a tendency to overlook the excellent dispersing action of soap and replace it with costly and less effective synthetic dispersing agents.

Dr. C. M. WHITTAKER: Instead of arguing about the use of soap as dispersing agent, more attention should be paid to the quality of the water used for dyeing.

Mr. C. P. ATKINSON: What are the conditions for the application of a crease-resistant finish to Courpleta blends?

Mr. BOULTON: In a blend of two-thirds Courpleta with one-third Fibro it should not be necessary to give a crease-resistant treatment. Staple fabrics containing a minor proportion of secondary acetate rayon are regularly crease-resisted in the trade. With any similar blend containing Courpleta there would be no difficulty in applying the same methods.

Mr. J. GREENWOOD: Courpleta seems to be a fibre with possibilities in bulking or texturing processes. Has anything been done in this field?

Mr. BOULTON: Not yet.

Mr. KRAMRISCH: Does Courpleta give rise to difficulties with regard to static electricity?

Mr. J. S. WARD: Courpleta yarns and staples are delivered with an antistatic finish. Should this finish be removed, the fibre may exhibit static effects, which are, however, reduced considerably in blends with hydrophilic fibres.

Dr. P. W. CUNLIFFE: Mr. Boulton has stressed the effect of heat on Courpleta and has shown that the fastness properties of dyes are controlled by the dyeing temperature or by subsequent heat-treatment. If the dyer is not aware of the particular end-use of the material, he could possibly supply better fastness properties with the same dye and the same material, e.g. by subsequent heat-treatment.

Mr. BOULTON: The question poses a general problem: the dyer of *any* fibre is often not aware of the particular end-use. In the case of Courpleta, dye fastness is improved both by using maximum dyeing temperatures and by any heat-treatments subsequent to dyeing.

Dr. CUNLIFFE: Has the lecturer any information on the course of shrinkage of wool–Courpleta blends, with decreasing Courpleta content?

Mr. BOULTON: It is known that quite small proportions of secondary acetate in wool blends reduce shrinkage considerably. In the case of Courpleta we have concentrated on the higher proportions, and to date full information on the relation between shrinkage and fabric composition has not been obtained.

Mr. J. T. MARSH: Is there any reason why Courpleta should not be produced at the same price as the secondary acetate?

Mr. J. P. STONEMAN: There are two reasons. Firstly, the solvent used is more expensive than that used in the spinning of secondary acetate; therefore solvent losses are of greater significance to the cost. Secondly, Seraceta is made on a much larger scale than Courpleta at present, and this has an obvious bearing on the economy of production.

Mr. L. MORRIS: Does Courpleta show any anomaly in blends; e.g. does it tend to form a surface layer, or conversely form the core of the yarn?

Mr. W. R. BEATH: Movement of any fibres during spinning depends upon staple lengths and deniers as well as the intrinsic mechanical properties of the fibre. Over the range of Courpleta blends already experienced, difficulties consequent on the differential movement of fibres have not been experienced.

Dr. J. M. PRESTON: Is the degree of crystallisation caused by heat-setting Courpleta affected by subsequent heat-treatments, as with nylon? For example, in the case of a pleated garment, can a heat-treatment, such as domestic ironing, be applied without affecting the degree of setting?

Mr. HADFIELD: With reference to Dr. Preston's question, what recommendations should be made to the finisher if it is known that the fabric is to be pleated? Would a mild setting treatment be adequate?

Mr. S. N. BRADSHAW: Deformation of Courpleta in pleating, embossing, etc. takes place under the combined influence of heat, pressure, and moisture. A heat-set fabric in which the Courpleta has become more crystalline may be further deformed at a lower temperature under the influence of pressure. The conditions required are, however, more severe than for the unset fabric. Therefore, when fabrics are set prior to pleating, temperatures greater than 180°C. (dry heat) or 115°C. (in steam) are not recommended.

Mr. K. MCLAREN: Which types of fading lamps were used in the assessment of the light fastness of Courpleta?

Mr. BOULTON: The discrepancies reported between values of light fastness measured in daylight and under a fading lamp were found using the Fade-Ometer. We are finding that results with a Fugitometer agree more closely with the results of daylight fading. The case of Courpleta with disperse dyes is another, and outstanding, example of the dangers of relying solely on results obtained with artificial light sources.

Mr. HADFIELD: Has there been any experience of "blinding" in the dyeing of Courpleta with azoic dyes?

Mr. WARD: No.

Mr. HADFIELD: Considering the differential dyeing of fabrics containing both triacetate and secondary acetate, has any difficulty been experienced in obtaining repeatable results?

Mr. WARD: The results obtained depend upon the particular dyes used and the time and temperature of dyeing. Skilful use of these variables enables the dyer to repeat patterns.

Mr. BRADSHAW: Dyers have for some time past been exploiting the differential dyeing behaviour of viscose and cuprammonium rayons to obtain similar results, from which it is evident that such methods are within the competence of a reasonably skilful dyer.

Mr. A. S. FERN: Is there any difference in dyeing properties, e.g. rates of dye diffusion, between the continuous-filament and staple forms of Courpleta?

Mr. WARD: The continuous-filament and staple forms of Courpleta are produced by essentially the same process, and consequently are very close in dyeing behaviour. We have not experienced any differences arising from rate of dyeing, but it must be remembered that the staple form is usually matt, and consequently will have a different appearance from the bright form when containing the same amount of dye.

Mr. J. A. CLARKE: If Courpleta fabric is subjected to a heat-treatment before dyeing, does any unevenness in this treatment result in irregularities in its subsequent dyeing behaviour?

Mr. BOULTON: One can heat Courpleta up to 180°C. without altering the dyeing properties; if temperatures above this are used, the dyeing rate is reduced, and any such heat-treatment should be applied uniformly.

Mr. A. E. WAYWELL: Has Mr. Boulton anything to say on the use of "carriers" in the dyeing of Courpleta?

Mr. BOULTON: Carriers provide additional problems for the dyer and we wish to avoid their use. We have not found it necessary to use them as a dyeing aid. Were it found necessary, a number of substances might be used to increase dyeing rate.

ERRATA

Third Report of the Fastness Tests Co-ordinating Committee

(J.S.D.C., 71, 283-342 (June 1955))

Page	§	Correction
283	(last paragraph)	*This should be reworded to read:* In five cases . . . *and a new line inserted:* 2 Washing Test No. 4 (Wool)— this is now withdrawn. *the remaining three tests 2–4 being renumbered* 3–5.
284	(first paragraph)	*The first line should read* . . . **seven** ISO tests . . . *and* Chlorinated Water *should be added.*
295	3.3 (Table I)	*The column headed "Bath 2" should read downward:* Bath 2 (for Natural and Regenerated Cellulose) – 3 5 – 0·1 11·5 80 176 1 30:1
296	5.6	*This should read:* Assess the effect on the unneutralised test-control specimen . . .
298	5.6	*This should read:* Assess the effect on the unneutralised test-control specimen . . .

Page	§	Correction
299	3.5	**Test Control**— A dyeing of 1·5% Solway Blue GS on wool cloth (see § 7.1)
	7.1	**Test Control**— A well wetted-out pattern of wool cloth is entered at 40°c. (104°F.) into a dyebath containing 1·5% Solway Blue GS, 10% sodium sulphate crystals ($Na_2SO_4.10H_2O$), and 3% sulphuric acid (sp.gr. 1·84), all percentages being calculated on the weight of the pattern, at a liquor ratio of 40 : 1. The dyebath is raised to the boil in 15 min., and boiled for a further 45 min. The pattern is then removed, rinsed in cold running tap-water, and dried.
307	1.1	This method is intended for assessing the resistance of the colour of textiles of all kinds and in all forms to the action of boiling water. It is mainly applicable to wool and textiles containing wool.
312	1.1	*This should read:* . . . colour of textiles of all kinds and in all forms to the action . . .
318	1.1	This method is intended for assessing the resistance of the colour of textiles of all kinds and in all forms to the action of soap and soda solutions used in the alkaline milling of wool.
	3.3	*Delete* or viscose rayon.
321	1.1 (Acid Felting)	*This should read:* . . . resistance of the colour of textiles in all forms to the action of acids used in the . . .
328	3.1.2	*This should read:* . . . and 0·1 g. tetrasodium pyrophosphate ($Na_4P_2O_7$, $10H_2O$) per litre . . .
340	NOTE	*This should read:* . . . Second Report except that the wash liquor is not changed after the first 10 min. and that assessments are now . . .

Notes

Election of Fellows

At the meeting of Council held on 13th July 1955 the following were elected Fellows of the Society—

Frank Leslie Barrett

(*Bolton, Lancashire; Director of the Research Laboratories, Bleachers' Association Ltd.*)

Frank Jordinson

(*Huddersfield; Lecturer in charge of Dyeing, Huddersfield Technical College*)

Alfred Ferdinand Kertess

(*Wilmslow, Cheshire; Director and General Manager, Stockport United Chemical Co. Ltd.*)

Frank Hubert Marsh

(*Leeds; Managing Director, Longclose Engineering Co. Ltd.*)

Meetings of Council and Committees

July

Council— 13th

Finance and General Purposes— 13th and 21st

Publications— 19th

Colour Index Editorial Panel— 5th and 21st

Fastness Tests Co-ordinating— 26th

Diplomas— 27th

Diplomas Executive— 7th and 9th

Perkin Main— 4th

Perkin Executive— 8th

Perkin Social— 19th

Bradford Technical College

Physicochemical Aspects of Dyeing

The Bradford Further Education Subcommittee in conjunction with the Yorkshire Council for Further Education is arranging post-advanced courses of lectures on (*a*) General Chemistry and Properties of Organic High Polymers, and on (*b*) Theories of Colour, Colour Measurement, and Colour Assessment. Course (*a*) will comprise 26 lectures held on Wednesday evenings at 7 p.m. beginning on Wednesday, 21st September 1955. Course (*b*), also comprising 26 lectures, will be held on Friday evenings at 7 p.m. beginning on Friday, 23rd September 1955. Both courses will be found of value to those preparing for the A.S.D.C. Examinations (Papers A and B). Completed application forms should be returned together with the fee of £1 10s. 0d. for each course to the Principal, Technical College, Bradford 7, Yorkshire, by 19th September 1955.

New Books and Publications

Wool

Its Chemistry and Physics

By P. Alexander and R. F. Hudson. Pp. ix + 404. London: Chapman & Hall Ltd. 1954. Price, 45s. 0d.

Anyone beginning the study of the chemistry and physics of wool keratin is at first appalled at the vastness of the topic, confused by the welter of details, and hampered in consulting the literature by the wide variety of journals in which it is scattered. This book, therefore, provides a useful collection of many references and systematically expounds most of the major problems.

Unfortunately, the book is like the barrel of honey containing a spoonful of tar: the honey is sweet and nutritious, but the taste of the tar pervades the whole barrel. A few major faults and little niggling errors and omissions spoil what is otherwise a valuable reference book. On p. 57, the assertion that only the Hooke's-law region is unaffected by the rate of applying stress is contradicted by the data of Sikorski and Woods (*Proc. Leeds Phil. Soc.*, **5**, 313 (1950)), and the statement that a wool fibre stretched 50% for 24 hr. takes a year to recover should obviously be that a fibre left stretched for a year completely recovered within 24 hr. when released in water. Fig. 8(*a*) on p. 66 does not represent the behaviour of ordinary wool but of supercontracted wool; the scale of extension should be negative to emphasise this. Finally, students must be warned that Fig. 7 on p. 367 wrongly gives the spacing between chains in α-keratin as 4·65 A. when it should be 9·3 A. and Fig. 8 (p. 368) similarly confuses the "backbone spacing" as that between three chains instead of two. The equation on p. 90 should be $v_a = (1 + a)/d_a$. There are several wrong references and some misprints.

In the chapter on "Sorption and Swelling", the symbol x is used for several different quantities, and this may lead to some confusion: it means an increase in pore size on p. 93, a particular vapour pressure on p. 100, and a length co-ordinate on p. 109. To use x for p/p_0 on p. 107 seems absurd when h is the symbol used for relative humidity on the very same page.

These errors do not detract from the magnitude of the work, which can be appreciated when it is realised that the book covers morphological and stereochemical structure, chemical composition and reactivity, mechanical properties, sorption, swelling, and diffusion as well as acid–base and dyeing equilibria. The price is high for a 400-page book, but despite the above-mentioned faults it is well worth buying (the second edition when corrected will be an even better bargain). All students and research workers in the field of keratin chemistry and physics will have to refer to it, since there is no other textbook available. The authors are to be congratulated on compiling such a monumental work after a mere seven years' acquaintance with the subject. L. PETERS

Chemistry and Chemical Technology of Cotton

Edited by K. Ward with contributions by 19 collaborators. Pp. xix + 782. New York and London: Interscience Publishers. 1955. Price, $20.00 or 160s. 0d.

Taken in general, this is an admirable book. Although specifically devoted to the subject of one variety only of textile fibre, the treatment of all the chemical technologies which serve to produce the finished textile material is so thorough and far-ranging that this could serve the student in technical college and university all through his student life, and could remain as a very valuable *vade mecum* when he takes up his industrial work.

Most of the chapters have been written by different hands, and although this may lead, at times, to a certain amount of overlapping, it is not objectionable. It means that each contributor has been able to go thoroughly into his own subject, to bring his own reading up to date, and to discuss certain out-of-the-way details that a single author might not have been able fully to appreciate. There are very few important references that have been overlooked, and in some subjects, notably sizing, desizing, and bleaching, there is information which has recently become available but which has now appeared for the first time in a comprehensive textbook.

The outlook differs, as might be expected, from one chapter to another. For instance, the chapters on dyeing and printing are rather strictly practical, with most of the emphasis on recipes and procedures, while that on mercerising is based much more on the investigated behaviour of the individual fibres in alkalis and other swelling agents. The latter is, in fact, a laboratory rather than a works contribution. Chapters on treatments to impart resistance to wetting, burning, and creasing do reflect very well many of the recent developments that have been occurring in these fields. A fair amount of space is devoted to chemical and performance testing and to a consideration of various deterioration processes which occur in textiles during their use. An interesting reflection of the importance of cotton yarns and fabrics as reinforcing materials in the rubber industry is provided by the inclusion of a special chapter to deal with the subject. As with most American books of this type, indexing is well done, and there is a useful glossary.

One of the least successful features in the section on dyeing is the rather poor quality of some of the sketches of dyeing machines and the inability to get down to modern ideas on the mechanisms of dyeing, although there is a subsection which claims to be devoted to this topic.

With a book planned on these ambitious lines, there are always likely to be some items which a reviewer may like to see expanded or modified, to coincide more closely with his own ideas or experiences. For instance, many might feel that

more copious illustration of a future edition would improve it and make the description of processes more graphic. In spite of its virtues this book is, one imagines, highly priced even for the American market. With an unfavourable rate of exchange it becomes prohibitively dear for the private buyer and also perhaps for many libraries. If it is to fulfil its purpose in this country the cost should be drastically reviewed.

H. A. TURNER

Cellulose Nitrate

The Physical Chemistry of Nitrocellulose, its Formation and Use

By F. D. Miles. Pp. xiii + 422. London: Oliver & Boyd. 1955. Price, 45s. 0d.

The author, well known for his work on cellulose nitrate, has aimed at the provision of a survey of the principles underlying the manufacture and uses of the polymer and the experimental work involved in their development and study. Written essentially for and from the standpoint of the physical chemist, this book includes much work hitherto unpublished or of limited accessibility.

The book deals first with the structure and the nitration of cellulose. Following a concise survey of the physical and chemical constitution of native and modified celluloses, which includes brief but useful accounts of microscopic identification and optical anisotropy, the subject of nitration is reviewed in detail, providing a very good account of technical processes, their basic theory, and such related aspects as denitration, heat of nitration, and the stabilisation of cellulose nitrates.

Subsequent chapters are concerned with physical and chemical properties of fibres and films of cellulose nitrate and of gels containing it. Structure and constitution, optical and mechanical properties, and light absorption are discussed, and heats of combustion and formation and other physical data are collected. Solvent absorption and gelatinisation are discussed in detail. Gels containing cellulose nitrate are divided into homogeneous, as used in the celluloid and leather-cloth industries, and heterogeneous, used in explosives.

The remainder of the book deals with the chemical reactions of cellulose nitrate and the properties of its solutions. Under chemical reactions are included thermal decomposition, hydrolysis, photochemical decomposition, cross-linking, and gelation. Successive chapters then deal with fractionation, with molecular weight determinations, and with solvents, solution, and solubility. In these frequent use is made of modern thermodynamic theories of polymer–solvent interaction and the physical chemistry of cellulose nitrate seen in relation to that of other high polymers. Solvation is discussed in detail, and its importance is clearly shown.

In view of the use of modern theories of polymer–solvent interaction, it is a pity that little or no mention is made of the more recent work on cellulose nitrate in this field. Thus light scattering, the application of modern hydrodynamic theories to the viscosities of dilute solutions, and more recent studies of fractionation and other aspects of cellulose nitrate–solvent interaction do not appear. While inclusion of the most recent work is not to be expected, it is surprising to find very few references to work published later than 1950. Apart from such omissions, few important aspects seem to have been omitted, although degradation in solution does not seem to be considered, and the Martin equation for extrapolation of viscosity data is not mentioned. Errors appear to be few. However, the membranes used in the Fuoss–Mead osmometer are not usually regarded as permeable to solute unless material of low molecular weight is present. Solute and solvent seem to be confused on p. 357.

These latter are minor criticisms, and the author is to be congratulated on providing, within the limits imposed by the omission of references to some of the more recent work, an authoritative and very useful account of the physical chemistry of cellulose nitrate, its formation, and its uses. It will be a welcome addition to the libraries of those concerned with cellulose derivatives and of polymer chemists generally. Readers of the *Journal* may find material of interest in the chapter on cellulose and in the treatment of polymer–solvent interaction, much of which is applicable to fibre–liquid interaction generally.

W. R. MOORE

Landolt–Börnstein
Zahlenwerte und Funktionen
aus Physik, Chemie, Astronomie, Geophysik, und Technik
Sechste Auflage
Band IV— Technik
Teil 1
Stoffwerte und mechanisches Verhalten von Nichtmetallen

Edited by E. Schmidt. Pp. xvi + 881. Berlin: Springer-Verlag. 1955. Price, DM 288.00.

The well known *Physikalisch-chemische Tabellen* of Landolt–Börnstein have been extended in the sixth edition to include technological data. Part 1 of Volume IV covers natural and artificial building materials and organic materials (wood, paper, pulp; fibres; plastics; natural and synthetic rubber).

The section on fibres (pp. 322–420) has been compiled by Prof. P. A. Koch, who has already published tables of data on the synthetic fibres in Continental periodicals. It covers the classification of fibres, their chemical structure, fine structure, molecular weight and degree of polymerisation, optical data, physical properties of the fibre substance (density, water uptake, electrical properties, etc.), fibre dimensions, and fibre strength data.

This collection of numerical data on fibres with full references to the original sources should prove to be of considerable value to those concerned with the scientific and technical aspects of textiles.

C. J. W. H.

Approved Methods for the Physical and Chemical Examination of Water

Revised 1953. Pp. ii + 59. London: Institution of Water Engineers. Price, 7s. 0d.

The Institution of Water Engineers in collaboration with the Royal Institute of Chemistry and the Society of Public Analysts and Other Analytical Chemists, having set up a Joint Committee to examine the question of standardising the methods to be used for the examination of potable water, published their recommendations in 1949. The second edition of this extremely useful report has now appeared and follows the same lines as the first edition.

A few major alterations may be noted. Thus the ethylenediaminetetra-acetate titration of hardness replaces the former method using palmitate, and the method for cyanide, thiocyanate and the F.A.S. (ferrous ammonium sulphate) residual chlorine test have been included in Appendix A.

The body of the book has been very thoroughly rechecked, as indicated by a number of minor amendments of details. The committee recommends that results be expressed in terms of "parts per million", and this bringing of English analysts into line with American practice is to be welcomed. The nomenclature to be used and the significant figures to be reported are given. It is, of course, most essential that water analysts should speak the same language, and the committee's recommendations for uniformity in the method of expressing analytical results should obviate what has been a fertile source of confusion in the past. It will, of course, produce a break of continuity in the methods and the mode of expression of results used in some laboratories, but the outcome should be entirely on the credit side.

Methods are given for the physical observations and determinations normally carried out on waters, also chemical methods of determination for chloride, hardness, free carbon dioxide, biological oxygen demand, oxygen absorbed, nitrate, nitrite, sulphate, phosphate, silica, chlorine, and all metallic ions normally of interest to water analysts.

Although the *o*-tolidine test for residual chlorine is not considered entirely satisfactory, the committee suggest that, in view of the state of flux existing on the subject of residual chlorine determinations, this test and the other methods which they give could well be regarded as standards for use in this country, but that future detailed study is required with a view to recommending a standard procedure.

The book is naturally a "must" for any laboratory dealing with water analysis. Although the list of contents is quite detailed, it appears to the reviewer that, despite the compactness of the book, an index would have been useful. This is, however, a very minor criticism compared with the overall usefulness of this valuable little volume.

J. A. RADLEY

Manual on Industrial Water

ASTM Special Technical Publication 148–A

Prepared by A.S.T.M. Committee D–19 on Industrial Water. 2nd printing 1954. Pp. viii + 420. Philadelphia: American Society for Testing Materials. Price, $ 5.00.

The present edition of this book sustains the excellence of the first edition, which was previously reviewed in these pages (J.S.D.C., **70**, 191 (1954)). It has been enlarged by the addition of the new and revised methods and several proposed methods published in 1953 and 1954.

Additions to the methods of analysis include a tentative scheme for industrial water and industrial waste water; chemical oxygen demand (dichromate); chlorine requirements and residual chlorine; non-referee methods for hardness, hydroxyl ion, nitrate and nitrite (tentative); odour, oily matter, pH value, and sulphides in industrial waste waters and sulphite in industrial waters.

The methods of testing which have been added include the determination of the thickness of deposits on tubular heat-exchanger surfaces and the evaluation of acute toxicity of industrial waste to fresh-water fishes.

The addition of a table of contents, itemised in much greater detail than before, another listing the A.S.T.M. standards relating to industrial water, and an index are very welcome in enabling much easier reference to be made to this excellent book.

J. A. RADLEY

Organic Reagents for Metals

and other Reagent Monographs

Volume I

Edited by W. C. Johnson. 5th edition 1955. Pp. viii + 199. Chadwell Heath: Hopkin & Williams Ltd. Price, 15s. 0d.

This volume contains twenty-seven monographs, some of which have already been published separately, while others have been extensively revised. The title of this edition has been expanded to cover reagents for certain acid radicals (nitrate and perchlorate) and organic functional groups (phenols and α-amino acids). One monograph describes the use of "sequestric acid" (ethylene diaminetetra-acetic acid) and its salts in the determination of the hardness of water, and another deals with the Karl Fischer reagent for water.

C. J. W. H.

Worsted Drawing and Spinning

Part I: Drawing

By H. Walker. Pp. vii + 239. Manchester: The Textile Institute. 1954. Price, 21s. 0d. ($ 3.00).

This book is divided into ten sections, covering open drawing, gill boxes, open drawing boxes, cone drawing, French drawing, Anglo-Continental drawing developments and production, and faults in stop motions.

C. J. W. H.

Mechanics for Textile Students

By W. A. Hanton. Pp. xi + 336. Manchester: The Textile Institute. 1954. Price, 25s. 0d. ($ 3.50).

This book is divided into three parts, dealing respectively with the study of movement, statics, and applications of force in causing motion. An appendix gives some useful formulae. The subject and examples are related directly to the experience of most students of textile technology.

C. J. W. H.

Abstracts from British and Foreign Journals and Patents

The titles of abstracts may be modified. Abbreviations of names of firms are listed in J.S.D.C., **68**, *23 (Jan. 1952), and also, together with symbols and the periodicals abstracted, in the annual index*

I—PLANT; MACHINERY; BUILDINGS

PATENTS

Mixing, Vacuum Drying and Grinding Disperse Dyes. Struthers Wells Corpn. *USP* 2,677,504

Granulation of Carbon Black and other Finely Divided Solids. Deutsche Gold- und Silber-Scheideanstalt vormals Roessler. *BP* 732,160

Carbon Black. Columbian Carbon Co. *USP* 2,680,061

Improved furnace for making carbon black by heating gaseous hydrocarbons at temperatures above that at which they are decomposed to carbon black, there being no limitations to the width of the furnace nor with respect to the relative volumes and mass velocities of both the make gas and the blast flame gas. C. O. C.

Spreading Fibre Masses Evenly on Conveyors. Courtaulds. *BP* 730,815

Fibres which are deposited in irregular masses on a conveyor are spread into a layer of even thickness by a spreader, which comprises a shaft, at right angles to the conveyor, to which are fixed a number of inclined discs rotating against the direction of the conveyor (in appearance it is similar to an agricultural disc-harrow). The uniform layer emerges in a state more suitable for continuous liquid treatments. J. W. B.

Apparatus for Removing Liquid from a Wet Running Yarn. Celanese Corpn. of America. *BP* 728,778

Apparatus is described for removing liquid from a running yarn, especially a freshly-formed wet-spun yarn, without damaging the yarn. W. G. C.

Conditioning Yarn during Winding. Kearny Manufacturing Co. *USP* 2,678,024

Dyeing, Sizing, and Drying Warps in Open Width. Bachmann Uxbridge Worsted Corpn. *USP* 2,679,086

Carrying Materials through Liquors. DuP. *BP* 729,804

Rotary carriers support a continuous length of the material and carry it through the liquors. Gravitationally induced motion of the supporting bars during rotation of the carrier passes forward the material supported by the bars. No part of the material remains in contact with any support for an appreciable number of rotations. C. O. C.

Drying Warps in Open Width. Bemberg. *BP* 731,415

Guide Control for Fabric, Paper, etc. Licentia Patent-Verwaltungs-G.m.b.H. *BP* 730,447

The fabric, etc., travels over a support which is adjustable across the direction of movement of the material. The adjusting movement of the support is operated by a constantly running motor through two magnetic powder clutches, one for adjustment on the left and the other for adjustment to the right. The energising current for either clutch is automatically switched on upon deviation of the fabric to one or the other side. C. O. C.

Tenter. B. Kawaguchi. *BP* 730,440

A tenter in which the chain travels alternately up and down vertically in a zig-zag path. It enables drying to be carried out in absence of warp tension and with considerable economies in both heat and time. C. O. C.

Spreading Tubular Fabric. Proctor & Schwarz. *USP* 2,680,279

Apparatus accommodating a wide range of spreader sizes, to each size of which it is adjustable. It both supports the spreader and pushes the fabric over it. C. O. C.

Calender. Armstrong Cork Co. *USP* 2,678,464

An adjusting screw mechanism permits alignment of the roll bearings while one of the rolls is being moved during roll adjustment. C. O. C.

Cylinder Dryer. Messinger Bearings. *BP* 730,637

A cylinder dryer whose structure is such that irregularities and imperfections caused by expansion and contraction of the machine during operation are avoided. *BP* 730,698

The steam or other heating fluid is introduced into the cylinders so as to provide uniformity of temperature to the cylinders' surfaces. C. O. C.

Maintaining Constant Web Tension during Winding. James Halley & Sons. *BP* 730,412

Marking Device for a Cloth or Threads passing through a Machine. Gebrüder Sucker. *BP* 730,556

Screen Printing Table. M. Stoffel. *BP* 731,032

A steel tape attached to two opposing sides of a rectangular frame and extending over the entire length of the frame forms the support for the fabric to be printed. The amount of tension put on to the tape controls the softness of the printing table. C. O. C.

Velour Finish on Hat Felts. J. Schweig. *BP* 731,493

The hat felt is rotated against a rotating grinding wheel. This wheel moves along a rigid guideway parallel to the straight side portion of the nearer horizontal generatrix of the felt to the base of the felt. It then moves along the arcuate end of the same generatrix and returns along the same path. When the end of the felt is curved, the grinding wheel preferably moves pivotally along the curve about a vertical axis extending through the centre of curvature of the curve. C. O. C.

Tenter Frame Washing System of Mercerising Ranges (X p. 483).

Shrinking Woollen and Worsted Fabrics (X p. 483).

II—WATER AND EFFLUENTS

PATENT

Grease Recovery from Scouring Liquors. Pacific Mills. *BP* 730,250

Wool scouring effluent is permitted to stand for specified times before clarifying to recover the grease, the effluent from the recovery being again allowed to stand, after a preliminary heating and addition of common salt, before a further clarification. The final effluent is suitable for returning to the first scouring bowl. J. W. B.

III—CHEMICALS; AUXILIARY PRODUCTS; FINISHING MATERIALS

Critical Micelle Concentrations of Potassium Alkyl Malonates in Aqueous Solution. K. Shinoda. *J. Physical Chem.*, **59**, 432–435 (May 1955).

Critical micelle concentrations have been determined by the visual colour change of dye, absorption spectrum of dye, solubilisation, and surface tension methods. The logarithm of the critical micelle concentration is linearly related to the number of carbon atoms in the chain. It is also linearly related to the logarithm of the total concentration of added counter-ions. W. R. M.

Critical Micelle Concentration of Sodium Lauryl Sulphonate. R. J. Williams, J. N. Phillips, and K. J. Mysels. *Trans. Faraday Soc.*, **51**, 728–737 (May 1955).

The critical micelle concentration of sodium lauryl sulphonate in water and in sodium chloride solutions has been determined by conductivity, dye solubility, and light-scattering studies. Results obtained by these methods are in agreement. The critical micelle concentration is found to vary with a power of the concentration of the counter-ions. W. R. M.

Heat of Oxidation of Sodium Formaldehyde-sulphoxylate. A. N. Derbyshire and R. Thornton. J.S.D.C., **71**, 244 (May 1955).

Air oxidation of 1 g. of Formosul is accompanied by the evolution of about 1 kcal., so that oxidation of only about one-tenth of the Formosul in a typical print-paste would produce a rise in temperature of the order of magnitude of those observed in a steamer. C. J. W. H.

Fixation of Vegetable Tannins by Polyvinylpyrrolidinone. K. H. Gustavson. *Svensk Kem. Tidskr.*, **66**, 359–362 (1954): *Chem. Abs.*, **49**, 7278 (25 May 1955).

Polyvinylpyrrolidinone (I) precipitates both condensed and hydrolysable types of tannin from solution. The latter type (tannic acid) is precipitated at pH 3·4 in the amount of 80–90% of I, but the precipitation of the first (wattle tannin) is independent of pH from 2 to 7, being precipitated in the amount of 90–100% of I. Precipitation does not occur at pH > 8. The fixing power of I is caused by the mesomerism of the C(:O)·N: group and shows that the imino H of the keto-imide group is not essential for fixation. The sensitivity of I for tannins is about 0·1 that of gelatin. Non-ionic Cr compounds are not precipitated by I but colloidal silicic acid is. C. O. C.

PATENTS

Wetting Agents. DH. *BP* 730,461

Addition of a water-insoluble neutral trialkyl or trialkoxyalkyl phosphate, e.g. tri-*n*-butyl or tributoxyethyl phosphate, to known wetting agents, e.g. di-*iso*propylnaphthalene sulphonic acid, sodium alkylaryl sulphonates, sodium lauryl sulphate, Invadin N, etc., greatly increases their wetting power. C. O. C.

High Molecular Weight Polymer Detergent. Wyandotte Chemicals Corpn. *BP* 731,603

Propylene oxide is condensed with an organic compound containing > 1 reactive H atom, e.g. propylene glycol, to form a polyoxypropylene polymer of average mol. wt. < 900 (determined by hydroxyl number). This polymer is then condensed with 10–80% of its wt. of ethylene oxide. The products are solid non-ionic detergents. C. O. C.

Detergent. Colgate-Palmolive Co. *USP* 2,679,482

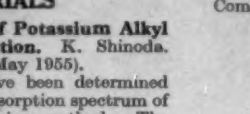
Addition of small amounts of aliphatic glycols containing a straight chain of 10–20 C, and having adjacent terminal hydroxy groups, e.g. hexadecane-1:2-diol, to sulphated and sulphonated detergents imparts improved foaming and/or detergent properties. C. O. C.

Detergent Compositions. BrC. *BP* 730,572

Aq. soln. of anion-active detergents, e.g. alkali metal alkyl sulphates, are thickened by incorporating a methyl cellulose of $< 30\%$ methoxyl content and of viscosity > 750 centipoises in 2% soln. at 20°C. Presence of $> 5\%$ of a salt in the detergent is permissible. In one example a 3% aq. soln. of methyl cellulose (32·7% methoxyl, viscosity 1418 c.p.s.) is added at room temp. to 5 times its wt. of Teepol 530, with stirring. A clear solution of 1% of Na_2SO_4 raised the viscosity to 270 c.p.s., and the solution does not coagulate on dilution with water. J. W. B.

Cationic Surface Active Agents. S. A. Farmaceutici Italia. *BP* 730,394

Compounds of formula—



(R = straight chain Alk of 8–16 C; A = acid radical of a strong acid, e.g. HCl), e.g. *n*-propylether of 2:4-diguanidinophenol dihydrochloride, are detergents having bactericidal properties. C. O. C.

Polyoxyalkylene Surface Active Agents. Wyandotte Chemicals Corpn. *USP* 2,677,700

Methods of making compounds of formula—

$$Y(CR^1R^2{\cdot}CR^3R^4{\cdot}O)_nX$$

(Y = residue of an organic compound containing a single H atom capable of reacting with a 1:2-alkylene oxide; R^1, R^2, R^3 and R^4 = same or different, H, aliphatic or aromatic radical, at least one of them not being H; $n > 6{\cdot}4$ as determined by hydroxyl numbers; X = a water-solubilising group). C. O. C.

Spinning Oils Containing Surface Active Agents. Scottish Oils. *BP* 730,886

A spinning oil composition comprises a metal salt of a sulphated olefin of b.p. < 180°C. (e.g. 29·5 parts by wt. of aq. sol. containing 20% active agent content), a mineral oil, e.g. Venezuelan Spindle Oil (59·0), a fatty acid of > 11 C (e.g. 9·5 parts of 70% saponifiable oleine) and either diethylene glycol monobutyl ether or normal butyl alcohol (2). J. W. B.

Alkoxy Alkylene Pyridinium Chlorides. Onyx Oil & Chemical Co. *USP* 2,681,342

A tertiary amine is neutralised with conc. HCl and the mixture heated to drive off all the water and unreacted pyridine. The cooled pyridinium hydrochloride is treated with a lower aliphatic alcohol and paraformaldehyde. The product is then freed from by-products and excess reactants. Thus $\beta\gamma$-picoline is neutralised with conc. HCl, heated to 200°C. and the anhydrous salt treated with methanol and paraformaldehyde. After removal of untreated methanol and the methylol obtained as a byproduct a mixture containing methoxy methylene $\beta\gamma$-picolinium chloride is obtained. Such products are used as emulsifying agents and for rendering cellulosic fabrics dimensionally stable. C. O. C.

Sizes. Atlas Powder Co. *BP* 731,260

An aqueous solution containing 5–15% of starch and 0·05–2·0% of a polyoxypropylene ether-ester of a higher fatty acid, e.g. polyoxyethylene ether-ester of stearic acid containing 8 polyoxyethylene groups, is a highly efficient warp size. C. O. C.

Milling Agents. Metallgesellschaft. *BP* 731,243

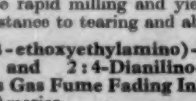
Organic orthophosphates containing at least one hydrocarbon radical of > 5 C, e.g. mono*cyclo*hexyl phosphate, are excellent milling agents superior to soap or sulphuric acid. They give rapid milling and yield cloths of better handle and resistance to tearing and abrasion. C. O. C.

2-Anilino-4-(β-ethoxyethylamino)-6-amino-1:3:5-triazine and 2:4-Dianilino-6-amino-1:3:5-triazine as Gas Fume Fading Inhibitors. Celanese Corpn. of America. *BP* 729,789

These two compounds are excellent gas fume fading inhibitors having good affinity for cellulose acetate or other organic derivatives of cellulose so that they may be added to the dyebath. C. O. C.

Di-(dialkylaminoalkyl)phthalates as Gas Fume Fading Inhibitors. Celanese Corpn. of America. *BP* 731,007

Compounds of formula—



(R^1 and R^4 = same or different alkylene; R^2, R^3, R^5 and R^6 = same or different Alk), e.g. di(diethylaminoethyl)-phthalate, are excellent gas fume fading inhibitors. They are applied as an aqueous dispersion in the pad. C. O. C.

Aqueous Textile Finishing Composition containing Methylol High Fatty Acid Mono- or Diamides. DuP. *USP* 2,676,936–7

Durable water-repellency and dimensional stability are imparted by use of aqueous dispersions of methylol fatty acid amides, polyvinyl alcohol and, preferably, a water-soluble heat-setting amide–formaldehyde resin or of methylene fatty acid diamides, polyvinyl alcohol and, preferably, a water-soluble low alkyl ether of a methylol melamine resin. C. O. C.

Solutions of Acrylonitrile Polymers or Copolymers. Vereinigte Glanzstoff-Fabriken. *BP* 731,789

Addition of 5–20% compounds of formula—

$$CH_3(CH_2)_nCO{\cdot}O{\cdot}R{\cdot}O{\cdot}CO{\cdot}(CH_2)_nCH_3$$

(R = CH_2 or $>CH{\cdot}CH_3$; n = 0, 1 or 2) to dimethylformamide used for dissolving acrylonitrile polymers or copolymers prevents undue swelling of the polymer during dispersion and prevents any tendency towards discoloration. C. O. C.

Polymeric Zirconium Compounds. DuP. *USP* 2,681,922

The products obtained by treating a compound of formula—

$$Zr(OR)_4$$

(R = Alk, *cyclo*Alk, Ar or aralkyl) is treated with $\leq 0{\cdot}5$ mol. of an anhydrous monocarboxylic acid, e.g. 1 mol. tetraethylzirconate and 0·5–4·0 mol. stearic acid for 1 hr. at 50–112°c., are useful as water-repellent agents for textiles, dispersing agents for pigments, milling aids for pigments, especially for phthalocyanines, etc. C. O. C.

Flame-resisting Agents. BASF. *BP* 731,130

Aqueous dispersions containing emulsifying agents, of difficultly volatile chlorinated hydrocarbons of Cl content $\leq 30\%$ by wt. and which contain in the continuous phase a water-soluble polymer, e.g. polyacrylic amide, and in the continuous and/or disperse phase inorganic flame-resisting agents, e.g. Sb_2O_3 and $CaCO_3$, are very effective and cheap flame resisting agents. C. O. C.

Flame-resisting Coating Compositions. Monsanto. *USP* 2,680,077

A flame-resisting composition of great stability consists of a combination of a compound containing free OH groups with the polymers obtained by condensing ammonia with phosphoryl chloride. Such compositions, e.g. a mixture of Glyptal (a phthalic anhydride-glycerol-resin) and 10% of polyphosphorylamide, when applied to wood or other combustible material, yield a film which intumesces when treated with a flame, the resulting porous heat-insulating char protecting the wood from the flame. C. O. C.

Fire-retardant Coating Composition. Sherwin-Williams Co. *USP* 2,681,326

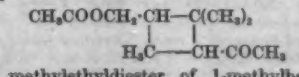
A coating composition which on activation by flame yields a voluminous intumescence consists of (1) an alkaline catalysed urea derivative aldehyde resin, (2) an acidic ammonium phosphate catalysed condensate of an aldehyde and cyanamide, dicyandiamide or guanidine, (3) an inorganic ammonium salt which yields ammonia on heating, (4) a saturated aliphatic dicarboxylic or hydroxy-dicarboxylic acid and (5) china clay and zinc oxide. C. O. C.

Light Coloured Syntans of Good Fastness to Light. American Cyanamid Co. *USP* 2,677,675

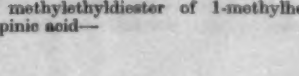
Presence of $\leq 0{\cdot}5$ part per thousand of free SO_3 in nuclearly sulphonated phenol-formaldehyde condensates imparts protection against discoloration upon storage. C. O. C.

Pinonic Acid Derivatives as Fungicidal Plasticisers for Cellulose Esters. T. Hasselstrom. *USP* 2,679,461

Methylpinonate



and the methylethyldiester of 1-methylhomodehydroethylene pinic acid—



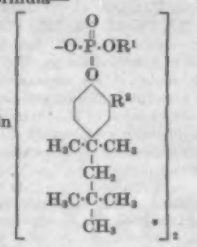
are excellent fungistatic plasticisers for cellulose esters. C. O. C.

Insect Repellent Compositions. Dow Corning Co. *USP* 2,681,878

Incorporating an organopolysiloxane in an insect repellent greatly increases the period of effectiveness. C. O. C.

Tin Ester Salts of Phosphoric Acid. ICI. *BP* 731,386

Compounds of formula—



(R^1 = Alk of 13C; R^2 = H or Alk), e.g. stannous *p-tert.*-octyl-phenylmethylhydrogen phosphate, dissolved in organic liquids form vehicles for paints or inks. Added to liquid hydrocarbons used for dry cleaning, they prevent re-deposition of soil on the goods and they are also useful as corrosion inhibitors for ferrous metals. C. O. C.

Dyeing Mixtures of Animal Fibres and Nylon— Reserving Agents for Nylon (VIII p. 481).

IV—RAW MATERIALS; INTERMEDIATES; COLOURING MATTERS

Preparation of Aromatic Aldehydes by means of the Dinitrogen Tetroxide Reagent. B. O. Field and J. Grundy. *J.C.S.*, 1110–1112 (April 1955).

A variety of substituted benzyl alcohols are converted into the corresponding aldehydes by N_2O_4 in $CHCl_3$ or CCl_4 soln. The excellent yields (90–98%) and the simplicity of the technique show the reaction to be a valuable general preparative method for aromatic aldehydes. H. H. H.

Nitration of 1:2-Diazo-oxides of Naphthalenesulphonic Acids. L. Blangey. *Helv. Chim. Acta*, **38**, 744–752 (May 1955).

The 7- and the 8-sulphonic acids are shown to lead to the 6-nitro deriv. in practically quantitative yield. The nitration of the 6-sulphonic acid resulted in the 8-nitro cpd. (88% yield) together with 6-nitronaphthalene-1:2-diazo-oxide (6·7%). Several cpd. are prepared in orientating the products, among them 6-amino-2-naphthol-7-sulphonic acid. H. E. N.

Orientation Effects in the Diphenyl Series. XIV—Thermal Decomposition of some "Diazoperbromides". S. M. Loh and E. E. Turner. *J.C.S.*, 1274–1277 (April 1955).

The bisdiazonium perbromide derived from 2-nitrobenzidine decomposes in glacial acetic acid at 95°c. to give 2′:4:4′-tribromo- together with about twice its weight of 4:4′-dibromo-2-nitrodiphenyl. It would appear that the diazonium group attached to the ring containing the NO_2 group is less stable than the other diazonium group, so that bromination leading to the tribromonitro cpd. occurs while the ring not containing the NO_2 group retains the (*meta*-directing) diazonium grouping; only in this way can the 2′-bromination be interpreted. H. H. H.

Reaction of Diazo Compounds with Sulphamic Acid and its Derivatives. III—Hydrolytic Decomposition of 1-Aryl-3-methyl-3-triazensulphonic Acids. D. Z. Zavel'skiĭ and L. A. Lishnevskaya. *J. Gen. Chem. U.S.S.R.*, **25**, 362–371 (Feb. 1955).

Hydrolytic decomp. of 1-aryl-3-methyl-3-triazensulphonic acids generally results in loss of the sulpho group, and subsequent breakdown of the arylmethyltriazen formed

yields nitrogen, methanol, and an arylamine. Other products, e.g. the symmetrical diaryltriazen, may be formed as a result of cleavage with formation of a diazonium salt (cf. J.S.D.C., **71**, 416 (July 1955)).

A. E. S.

Synthesis of some Pyrazolones and Pyrazolex. J. Büchi, H. R. Meyer, R. Hirt, F. Hunziger, E. Eichenberger, and R. Lieberherr. *Helv. Chim. Acta*, **38**, 670–679 (May 1955).

A series of alkylated and dialkylaminoalkylated cpd. are prepared for pharmacological examination. H. E. N.

Radioactive Fast Yellow. O. Hromatka, L. Stentzel, and E. Broda. *Mh. Chem.*, **86**, 137–140 (Feb. 1955).

The sodium salt of 1′-^{14}C-4-aminoazobenzene-3:4′-disulphonic acid is synthesised in 69·3% yield from 1-^{14}C-aniline hydrochloride. It is separated from Na_2SO_4 by extraction with absolute alcohol and from monosulphonate by the stronger adsorption of the latter on cellulose powder. 92% of the radioactivity is lost in the rearrangement of active diazoaminobenzene in presence of inactive aniline and aniline hydrochloride. H. E. N.

Organic Pigments. VII—Colour of Bisnitrophenylamine Derivatives. E. Koike and H. Iida. *J. Chem. Soc. Japan, Ind. Chem. Sectn.*, **57**, 546–550 (1954); **VIII— Rôle of Impurities in the Raw Material on the Pigment of Chlorophenylazonaphthol.** E. Koike, S. Suzuki, I. Tanakadata, and N. Kuwabara. *Ibid.*, 550–552; **IX— Preparation of Pigments from Copper Phthalocyanine.** E. Koike and T. Yagi. *Ibid.*, 552–555; *Chem. Abs.*, **49**, 7260 (25 May 1955).

VII— Spectrophotometric measurements of the colour of 24 purified bisnitrophenylamine derivatives as powder, 1% solution in pyridine, and dyed on acetate rayon indicate that they correspond to the colours of very thin, medium thick, and thick liquid layers. The principal absorption wavelength of the solution increases linearly with decrease in transparency. Similarly reverse parallel relationships were seen between the principal wavelength of both the powdered dye and dyed acetate rayon and the reflection index.

VIII— The maximum amount of 1-naphthol and *o*-chloroaniline that may be present in the 2-naphthol and *p*-chloroaniline used is 0·1% and 0·5% respectively if a pigment of reasonably fine colour is to be produced.

IX— The best method of preparation is to prepare a soln. of 1 part of copper phthalocyanine in 8 parts of 92·4% H_2SO_4, precipitate by diluting with a large volume of water, and stir, filter, and wash repeatedly. C. O. C.

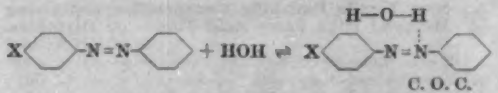
Direct Cotton Dyes derived from Cyanuric Chloride. III— High-molecular Dyes. H. Iida and K. Yamakawa. *J. Chem. Soc. Japan, Ind. Chem. Sectn.*, **57**, 587–589 (1954); *Chem. Abs.*, **49**, 6609 (10 May 1955).

A description of the preparation of three dyes— (1) from 1 mol. of cyanuric chloride, 2 mol. of H acid, and 1 mol. of aniline, followed by coupling with 2 mol. of diazotised *p*-aminoacetanilide and finally hydrolysing; (2) from 1 mol. of cyanuric chloride, 2 mol. of aniline, and 2 mol. of diazotised 2-amino-5-nitrobenzoic acid and finally reducing the nitro group; (3) from 1 mol. of cyanuric chloride, 2 mol. of J acid, 1 mol. of aniline, and 2 mol. of 2-amino-5-nitrobenzoic acid and finally reducing the nitro groups.

C. O. C.

Relationship between Absorption Spectra and Chemical Constitution of Dyes. XXVIII— Hydration of Azo Dyes in Organic Solvents. W. R. Brode, I. L. Sheldin, P. E. Spoerri, and G. M. Wyman. *J. Amer. Chem. Soc.*, **77**, 2762–2765 (20 May 1955).

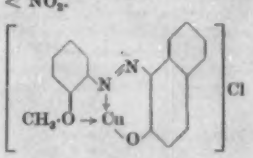
Addition of water to alcoholic solutions of 4-amino-, 4-dimethylamino-, 4-hydroxy-, and 4-methoxy-azobenzenes results in formation of a new absorption band at a somewhat longer wavelength than that of the main absorption band in the visible region, indicating formation of some new species. This new band seems to be caused by formation of a hydrogen bond between the azo group and water—



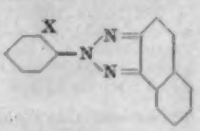
C. O. C.

Internal Complexes of Azo Compounds. I— Complexes formed by Copper with some *o*-Monohydroxyazo Compounds. II— Reaction of Copper Salts with some *o*-Monoaminoazo Compounds. V. I. Mur. *J. Gen. Chem. U.S.S.R.*, **24**, 572–574 (March 1954); **25**, 374–380 (Feb. 1955).

It has been generally found that *oo′*-disubstituted azo cpd. in which the substituents are salt-forming groups (OH, NH_2, COOH) yield 1 : 1 complexes with cupric salts (1 Cu atom per mol. of azo cpd.), whereas *o*-monosubstituted cpd. yield 1 : 2 complexes. These findings are confirmed for 1-*o*-hydroxyphenylazo- and 1-*o*-carboxyphenylazo-2-naphthols, and a 1 : 1 copper complex is prepared also from 1-*o*-(carboxymethoxy)phenylazo-2-naphthol. However, under the same conditions (reaction with $CuCl_2$ in alcoholic soln.), 1-*o*-methoxyphenylazo-2-naphthol and 4-*o*-methoxyphenylazo-3-methyl-1-phenylpyrazol-5-one also yield 1 : 1 complexes (e.g. I); they give reactions for Cl^-. These are converted into the 1 : 2 complexes previously reported when treated with aq. NH_3. The corresponding *o*-chloro and *o*-nitro cpd. form only 1 : 2 complexes. Analogous experiments are carried out with aminoazo cpd. derived from 2-naphthylamine and from 5-amino-3-methyl-1-phenylpyrazole. Analogous 1 : 1 complexes of the *o*-methoxyphenylazo cpd. are obtained, and these react analogously with ammonia. In general, complex formation with aminoazo cpd. is accompanied by oxidation with formation of a triazole (II). With respect to their effects on the tendency for this reaction to occur, *ortho* substituents (X) may be placed in the order $O{\cdot}CH_3 < CH_3 < Cl < NO_2$.



(I)



(II)

A. E. S.

Absorption Spectra and Molecular Structure. III— Modern Electronic Theory, and the Colour of Arylhydrazones of Acridine-9-aldehyde. A. A. Kharkharov. *Izvestiya Akad. Nauk S.S.S.R., Otdel. khim. nauk*, 326–334 (March–April 1955).

Absorption-spectrum measurements are made on soln. of the hydrochlorides and free bases of the phenyl-, *p*-acetamidophenyl-, *p*-methoxyphenyl-, *p*-formylphenyl-, *p*-nitrophenyl-, 1-naphthyl-, and 2-naphthyl-hydrazones of acridine-9-aldehyde, and also on soln. of the hydrochlorides of the phenyl- and *p*-nitrophenyl-hydrazones of 2-acetamidoacridine-9-aldehyde (cf. J.S.D.C., **70**, 366 (1954)). The results are interpreted on the basis of electronic theory, which correctly predicts, for example, the varying effect of a given substituent according to its position in the molecule— introduction of an acetamido group into the molecule of the hydrochloride of acridine-9-aldehyde phenylhydrazone results in a hypsochromic shift of 50 mμ. when the group enters position 2 of the acridine ring system, but a bathochromic shift of 36 mμ. when it enters the *para* position of the phenyl group. It is emphasised that electronic theory does not displace pre-electronic theories of colour, but lends them precision and unifies them. A. E. S.

Tautomeric Compounds. XVIII— Reaction of 4-*iso*-Propylidene- and 4-(1-Arylethylidene)-3-methyl-1-phenylpyrazol-5-ones with *NN*-Dimethyl-*p*-nitrosoaniline and with Nitrous Acid. A. E. Porai-Koshits and M. S. Dinaburg. *J. Gen. Chem. U.S.S.R.*, **25**, 151–156 (Jan. 1955).

The condensation products formed by 3-methyl-1-phenylpyrazol-5-one with methyl ketones (see J.S.D.C., **71**, 192 (April 1955): formula I) react not only with aromatic aldehydes and diazo cpd. (yielding dimethin and azo dyes respectively, see *loc. cit.*), but also with aromatic nitroso

opd. with formation of the corresponding azamethin dyes. The latter, and the aldehydes formed by acid hydrolysis, may be converted into hydrazones by the action of arylhydrazines, and the products are found to be identical with the azo dyes obtained by direct coupling. Nitrous acid converts the aryl methyl ketone condensation products (*loc. cit.*: I, R^1 = aryl) into side-chain oximes (conversion of 1-arylethylidene into 1-aryl-2-(hydroxyimino)ethylidene), but it eliminates the ketone residue from the acetone condensation product (I, $R^1 = CH_3$) and yields 4-hydroxyimino-3-methyl-1-phenylpyrazol-5-one.

A. E. S.

Dyes having Antipyrin Nuclei. IV—Acid–Base Properties of Dyes. O. F. Ginzburg, D. V. Ioffe, and N. S. Mel'nikova. *J. Gen. Chem. U.S.S.R.*, **25**, 358–362 (Feb. 1955).

Further measurements (based on the change of colour with pH) of the hydrolysis constants of diantipyryiphenylmethane dyes show that they vary greatly (from 10^{-5} to 10^{-11}) as the substituents in the phenyl radical are varied (from *p*-$N(CH_3)_2$ to *p*-NO_2) (cf. J.S.D.C., **70**, 306 (1954)). New dyes having *m*-methoxy and 3:4-dimethoxy substituents are prepared.

A. E. S.

Preparation of *p*-Xylenol Blue. G. N. Kosheleva. *J. Appl. Chem. U.S.S.R.*, **28**, 307–310 (March 1955).

The preparation of the indicator *p*-Xylenol Blue (pH ranges: 1·2–2·8, red to yellow; 8·0–9·6, yellow to blue) by the condensation of 2:5-xylenol with *o*-sulphobenzoic acid in presence of zinc chloride, as given in the literature, gives very low yields (3·6%). The use of a mixture of phosphoryl chloride and zinc chloride as catalyst enables the pure indicator to be prepared in 71–73% yield.

A. E. S.

Indolenic Cyclisation of Ethylenic Colouring Matters—Resulting Indanic Colouring Matters. C. Dufraisse, A. Étienne, and B. Goffinet. *Compt. rend. Acad. Sci. Paris*, **235**, 1349–1352 (1952); *Chem. Abs.*, **49**, 6905 (25 May 1955).

The compounds obtained by cyclising 1:1:3-triphenyl-2-propen-1-ol in which at least one phenyl group at C-1 is substituted by $N(CH_3)_2$ have been synthesised by another method and shown to be 1:3-diphenylindenes. 3-*p*-Dimethylaminophenyl-1-phenyl- and 1:3-bis(*p*-dimethylaminophenyl)indene are colourless in acetic acid, whereas 6-dimethylamino-3-*p*-dimethylaminophenyl-1-phenyl- and 6-dimethylamino-1:3-bis(*p*-dimethylaminophenyl)indene give intensely blue solutions. Probably the indene adds an H ion to form a quinonoid cation, which, because its conjugation does not involve the substituent in position 1, is approx. the same colour with both compounds.

C. O. C.

Indophenols for Sulphur Dyes. IX—Kinetics of the Condensation Reaction between *p*-Nitrosophenol and *o*-Toluidine in Sulphuric Acid. O. Manabe, S. Moriwaki, and H. Hiyama. *J. Chem. Soc. Japan, Ind. Chem. Sectn.*, **57**, 520–523 (1954); **X—Kinetics of the Condensation Reaction between *p*-Nitrosophenol and Dimethylaniline or Diphenylamine in Sulphuric Acid.** *Ibid.*, 523–525: *Chem. Abs.*, **49**, 6609 (10 May 1955).

IX—Kinetic study of the reaction between *p*-nitrosophenol and *o*-toluidine at 0–10°C. showed the reaction to be of the second order for the concentrations of the two components. The velocity constant decreased sharply with concentration of H_2SO_4. Probably the reaction can be explained by assuming reaction between the free base of *o*-toluidine and the conjugated acid.

X—The velocity constant of the reactions between *p*-nitrosophenol and dimethylaniline or diphenylamine is of the second order for the concentrations of the respective components. The effect of H_2SO_4 concn. and the reaction mechanism are similar to those for the reaction between *p*-nitrosophenol and *o*-toluidine. In H_2SO_4 of the same concn. the velocity constant varies in the following order—diphenylamine > *o*-toluidine > dimethylaniline.

C. O. C.

Structure and Properties of Piazthiole. L. S. Éfros and R. M. Levit. *J. Gen. Chem. U.S.S.R.*, **25**, 199–210 (Jan. 1955).

Recent bond-length measurements (Luzzati, *Acta Cryst.*, **4**, 193 (1951)) indicate that piazthiole has the quinonoid structure shown, and not the benzenoid structure with quadrivalent sulphur. It is considered that the properties of piazthiole and its deriv., particularly the deep colour of its amino deriv., are in general accord with this view, but piazthiole is exceptionally stable for a quinonoid opd. and does not give addition reactions, e.g. with halogens. Nitration and sulphonation give the 4-monosubstituted opd.; chlorine in presence of a carrier gives 4:7-dichloropiazthiole. Other deriv. can be prepared from the corresponding *o*-phenylenediamine deriv. The aminopiazthioles give all the characteristic reactions of aromatic amines—they yield azo dyes when treated with diazonium salts; and they can be diazotised, if self-coupling reactions are avoided, and coupled with suitable components. The absorption spectra of some piazthiole deriv. are given.

A. E. S.

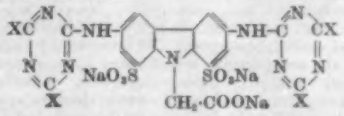
Preparation of 1:2:3:4:6:7:8:9-Octahydrodibenzothiophen. W. Cooper. *J.C.S.*, 1386 (April 1955).

The title opd. is conveniently obtained by direct reaction of S with *cyclo*hexanone or 2-*cyclo*hex-1′-enyl*cyclo*hexanone. The latter is probably an intermediate in the formation of the dibenzothiophen from *cyclo*hexanone.

H. H. H.

Syntheses of Organic Fluorescent Compounds. XXV—Synthesis of 9-Carboxymethyl-3:6-bis(4:6-disubstituted 2-*s*-triazylamino)carbazoledisulphonic Acids. Z. Yoshida, Y. Kato, and R. Oda. *J. Chem. Soc. Japan, Ind. Chem. Sectn.*, **56**, 411–413 (1953): *Chem. Abs.*, **49**, 6910 (25 May 1955).

Compounds of formula—

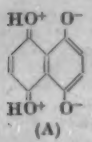


(X = $N(CH_3)_2$, $N(C_2H_5)_2$, $NH{\cdot}[CH_2]_2{\cdot}OH$, $NH{\cdot}C_6H_5$, $NH{\cdot}C_6H_4{\cdot}CH_3$-*o*, $NH{\cdot}C_6H_4{\cdot}CH_3$-*p*) were made by treating carboxymethyl-3:6-diaminocarbazoledisulphonic acid (I) with cyanuric chloride (II) and then substituting 2 Cl radicals in triazine groups with amines. The intermediate was obtained in pure form by titrating acetone solution of II into I at 0°C. All the compounds showed medium to strong blue to violet fluorescence under ultraviolet radiation.

C. O. C.

Quinones. V—Chemistry of Naphthazarin. D. B. Bruce and R. H. Thomson. *J.C.S.*, 1089–1096 (April 1955).

The synthesis, reduction, and addition reactions of naphthazarin (I) and of its 2-methyl, 2:6-dimethyl, 2:3- and 2:6-dichloro, and 6- and 7-chloro-2-methyl deriv. are described. In general I proves to be a relatively unreactive quinone, addition of Cl and aniline being very slow, HCl fails to react, while no definite compound results with HCN; on the other hand, toluene-*p*-sulphinic acid, toluene-*p*-thiol, and $KHSO_3$ react normally, the last two giving diaddition products, which indicate possible addition by a radical mechanism. Esterification and etherification of *peri*-OH groups have a marked effect on the additive powers of a naphthaquinone. In the past, the tautomeric nature of I has been emphasised, but it is evident that its general stability, intense colour, strong hydrogen bonding, and low chemical reactivity are due to the importance of resonance structures of the type A which lower the quinonoid activity of the mol. Further, the stability of the dianion is remarkable, the deep cornflower-blue soln. in aq. NaOH being stable for days. Reductions of the above naphthazarins with acid $SnCl_2$ give β-hydronaphthazarin (1:2:3:4-tetrahydro-5:8-dihydroxy-1:4-dioxonaphthalene and its deriv., except in the case of 3-chloro-2-methylnaphthazarin, where the Cl is not eliminated. Alizarin Black S (*C.I.*1019), a water-sol. opd. of I and $NaHSO_3$ and formerly described as a dark brown powder (*GP* 41,518), is found to be light yellow, and both acid and alkaline hydrolysis do not liberate I, indicating the opd. to be formed by the usual quinonoid 1:4-addition and not as a normal carbonyl bisulphite opd.



(A)

H. H. H.

Derivatives of Anthraquinone. I—Synthesis of 1-Amino-4-methoxyanthraquinone. T. Hayashi. *J. Chem. Soc. Japan, Ind. Chem. Sectn.*, **56**, 444–445 (1953): *Chem. Abs.*, **49**, 7251 (25 May 1955).

1-Methoxyanthraquinone was treated with a mixture of H_2SO_4 and HNO_3 at 5–10°c. to yield 1-nitro-4-methoxyanthraquinone. 12 g. of the product was mixed with ethanol (4 c.c.), water (58 c.c.), Na_2S (24 g.) and 25% NaOH (4 c.c.). The mixture was stirred for 1 hr., warmed in the water bath for 2 hr., poured into water (1 litre), filtered, washed, and dried at 110°c. 1-Amino-4-methoxyanthraquinone (10·1 g.) was obtained and was purified by recrystallisation from isoamyl alcohol. It had low affinity for saponified acetate rayon which it dyed red becoming dark red on exposure to light. C. O. C.

Hydrogen Bonding, and its Effect on Energy Distribution in Vibrational and Electronic Molecular Spectra. D. N. Shigorin and N. S. Dokunikhin. *Doklady Akad. Nauk S.S.S.R.*, **100**, 323–326 (11 Jan. 1955). **Hydrogen-bond Effects in the Vibrational and Electronic Spectra of Aminoanthraquinone Derivatives.** Idem. *Ibid.*, **100**, 745–748 (1 Feb. 1955).

Hydrogen bonds are of two types. Bonds of the first type—which occur intermolecularly, and sometimes intramolecularly—are essentially electrostatic; their formation will not greatly modify the electronic (ultraviolet and visible) absorption and fluorescence spectra. In bonds of the second type, which occur intramolecularly in conjugated systems, interaction with π-electrons of neighbouring atoms predominates over electrostatic effects, so that the bonding hydrogen must be regarded as a part of the conjugated system and as a determining factor in the electronic spectra; also, the polarity of the group containing the bonding hydrogen (O–H, N–H, etc.) will be reduced in such an intramolecular position, and its vibrational (infrared) band will have low intensity, long wavelength, and high diffuseness, as compared with the band for a similar group bonded intermolecularly. The truth of these and other inferences is illustrated in the first paper by reference to the properties of some anthraquinone deriv., particularly to their infrared spectra in the solid and vapour states (alone and as binary mixtures). Solid alizarin, for example, has a fairly sharp band at $\sim$ 3400 cm.$^{-1}$ (corresponding to the β-OH, which forms an intermolecular bond of the first type) and a very diffuse band at $\sim$ 3100 cm.$^{-1}$ (corresponding to the α-OH, which forms an intramolecular bond). Reference is made to the known difference in dissociation constant between the two OH groups of alizarin (α-OH, $K = 1{\cdot}1 \times 10^{-12}$; β-OH, $K = 6{\cdot}6 \times 10^{-9}$), to the difference in reactivity between α- and β-hydroxyanthraquinone deriv. in general, and to differences in their electronic spectra—the absorption and fluorescence spectra of the α-deriv. are displaced towards the longer waves and, whereas the fluorescence of the β-deriv. is quenched by O_2 and by NO, that of the α-deriv. is not. In the vapour state, i.e. at higher temp. and with greater intermolecular distances, intermolecular bonds are broken and intramolecular bonds are weakened; of the resulting changes in the spectra, the most noteworthy is the shift of bands due to intermolecularly bonded groups to positions, at shorter wavelength, corresponding to unbonded groups (a very sharp band at 3620 cm.$^{-1}$ for alizarin). The OH or NH group taking part in intramolecular bonding must be coplanar with the conjugated system, or a bond of the second type will not be formed. This fact is illustrated by comparing indanthrone (N–H in plane of ring system) and 1-methylaminoanthraquinone (non-planar arrangement), for which the respective N–H infrared bands are at 3165 cm.$^{-1}$ (diffuse) and 3300 cm.$^{-1}$ (sharp), the intensity of the latter being 10–12 times as high as that of the former.

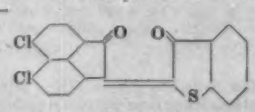
In the second paper, the comparison of planar and non-planar arrangements is extended to other substances. Planar arrangements are found, for example, in 1:4-di-*p*-toluidinoanthraquinone, 1-benzamido-4-chloroanthraquinone, and the parent 1:2-anthraquinone–acridone (naphth[2,3-*c*]acridine-5:14-dione), all of which behave like indanthrone. The second, non-planar group includes not only simple amino and methylamino deriv. of anthraquinone, but also deriv. of planar dyes in which the steric effects of substituents disturb the planarity of the molecule, e.g. 1:4-dimesidinoanthraquinone and 2:3-dimethyl-1:4-di-*p*-toluidinoanthraquinone. Although, as a result of non-planarity, the intramolecular hydrogen bond of 1-aminoanthraquinone is essentially of the electrostatic type and there is little difference between the infrared absorption spectra of 1- and 2-aminoanthraquinones, there is an appreciable difference, in the expected direction, between their electronic spectra. A. E. S.

Anthraquinone Derivatives. XXI—Hydrolysis of 1-Anthraquinonylmercury Chloride. XXII—Hydrolysis of 1-Anthraquinonylmercury Hydrogen Sulphate. V. V. Kozlov and B. I. Belov. *J. Gen. Chem. U.S.S.R.*, **25**, 410–416 (Feb.); 565–571 (March). **XXIII—Hydrolysis of Anthraquinone-1-sulphonic Acid.** V. V. Kozlov and A. A. Egorova. *Ibid.*, 809–814 (April 1955).

Kozlov has established that the sulphonation of anthraquinone in presence of Hg salts—as also the reverse, hydrolysis reaction—proceeds with intermediate formation of a mercury-containing anthraquinone cpd. It is now shown that the behaviour of 1-anthraquinonylmercury salts in sulphuric acid soln. of various concn. is in accord with the hypothesis that 1-anthraquinonylmercury hydrogen sulphate is the intermediate cpd. formed. It is shown also that anthraquinone-1-sulphonic acid is readily hydrolysed in presence of Hg salts; in their absence, hydrolysis can be effected only under very severe conditions. A. E. S.

Halogen Derivatives of Acenaphthenequinone. A. P. Karishin. *Ukrain. khim. zhur.*, **18**, 504–512 (1952): *Chem. Abs.*, **49**, 1682 (10 Feb. 1955).

Among the products of the oxidation of 3:4-dihalogenoacenaphthenes (*Chem. Soc.* numbering; the dibromo, dichloro, and bromochloro cpd. are used) are the corresponding 3:4-dihalogenoacenaphthenequinones and symmetrical 3:4-dihalogeno-8-oxo-7-acenaphthenylidene indigoid dyes (orange or brick red). 3:4-Dichloroacenaphthenequinone reacts with thionaphthen-3-ol with formation of the red dye—



A. E. S.

Polymethin Dyes. III—Vinylene-homologous Aldehydes of the Pyrrole Series. M. Strell and F. Kreis. *Chem. Ber.*, **87**, 1011–1018 (1954); **IV—Furyl-pyrryl- and Furyl-indolyl-methin Dyes.** M. Strell, A. Kalojanoff, and L. Brem-Rupp. *Ibid.*, 1019–1024; **V—An Extension of the Ehrlich Reaction.** M. Strell and A. Kalojanoff. *Ibid.*, 1025–1032: *Chem. Abs.*, **49**, 6935 (25 May 1955).

III—$HClO_4$ (2 c.c.) added in drops with stirring to $CH_3C_6H_4NCH{:}CHCHO$ (1·5 g.) and 2:4 dimethylpyrrole (I) in CH_3OH (50 c.c.) at -10°c. and stood for 2 hr. gave 3:5-dimethyl-2-pyrryl $CH{:}CHCH{:}NCH_2C_4H_2{}^+ClO_4{}^-$ (II). With $CH_3C_6H_4N(CH{:}CH)_2CHO$ (III) in a boiling water bath or when the pyrrole contained negative substituents, *sym*-dipyrrylpolymethin dyes were formed. Treatment of II with MgO in hot water, steam distilling off the $CH_3C_6H_4NH$, heating with animal charcoal and filtering hot yielded 3-(3:5-dimethyl-2-pyrryl)propenal which reacts with compounds containing reactive H to form unsym. polymethin dyes. A considerable number of similar syntheses are described.

IV—2:4-Dimethyl-3-ethyl pyrrole treated with $HClO_4$ and 2-(α-furyl)acrolein in glacial acetic acid yielded 1-(2-furyl)-3-(3:5-dimethyl-4-ethyl-2-pyrryl)trimethin perchlorate. A large number of similar syntheses are described. Plotting the absorption maximum of these polymethin compounds against the number of vinyl groups yields a straight line. For the furyl-indolyl compounds the band is

displaced toward the red and the intensity increased with increase in number of the vinyl groups. For trimethin compounds negative substituents in the β-position of the pyrrole ring displace the maximum toward the red while OH in the 2nd β-position displaces the maximum towards the blue. The more highly coloured compounds produced with furylpyrrylmono- and tri-methins and $C_6H_5NH_2$–$C_6H_5NH_2$,HCl probably result from cleavage by $C_6H_5{\cdot}NH_2$ to form $C_6H_5NHCH{:}CHCH{:}COH(CH{:}CH)_n{\cdot}$ $CH{:}C{\cdot}CH{:}CH{\cdot}C{:}NHX$ ($n = 0$ or 1).

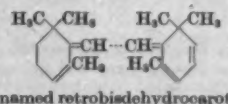
V— p-$(CH_3)_2NC_6H_4CH{:}CHCHO$ can be used to detect pyrroles having either the α- or the β-position free. The dyes obtained by treating this compound with $HClO_4$ and appropriate α- or β-free pyrrole or β-free indole are described. C. O. C.

Relative Stabilities of Chelate Compounds of Pyrrole Pigments. A. H. Corwin and M. H. Melville. *J. Amer. Chem. Soc.*, **77**, 2755–2759 (20 May 1955).

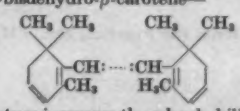
Increasing the electron-attracting power of methenes and porphyrins increases their affinity for copper and decreases that for magnesium. C. O. C.

Bisdehydrocarotenes. L. Zechmeister and F. J. Petracek. *J. Amer. Chem. Soc.*, **77**, 2567–2568 (5 May 1955).

The deeply coloured dehydrogenation product of β-carotene, "bisdehydro-β-carotene" has been shown to have the formula—



and has been renamed retrobisdehydrocarotene. Compared with 3:4:3′:4′-bisdehydro-β-carotene—



the retro structure increases the adsorbability and involves extensive fine structure in the main spectral band. Both of these compounds may result from direct dehydrogenation of carotenes. C. O. C.

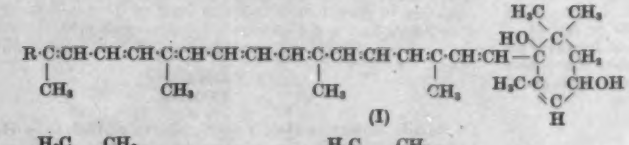
Carotenoid Syntheses. XV— Crystalline DL-α-Carotene. C. H. Eugster and P. Karrer. *Helv. Chim. Acta*, **38**, 610–612 (May 1955).

Preparation and properties are described.

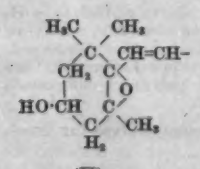
XVI— Stereoisomeric 1:3:7:12:16:18-Hexaphenyloctadecanonaenes. H. H. von Ziegler, C. H. Eugster, and P. Karrer. *Ibid.*, 613–638. H. E. N.

Trollixanthin and Trollichrome. M. Lippert, C. H. Eugster, and P. Karrer. *Helv. Chim. Acta*, **38**, 638–642 (May 1955).

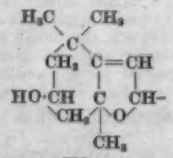
The former is isolated from the flowers of *Trollius europaeus* and rearranges into the latter on treatment with HCl–$CHCl_3$. Most probably trollixanthin has the structure I with R = II, whereas for trollichrome R = III.



(I)



(II)



(III)

H. E. N.

Colouring Matters of the Aphididae. XIV— The Course of Substitution Reactions and the Stereochemistry of the Erythroaphins. B. R. Brown, A. Calderbank, A. W. Johnson, J. R. Qualo, and A. R. Todd. *J.C.S.*, 1144–1153 (April 1955).

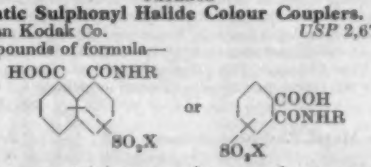
Solubility of Prussian Blue. M. Kohn. *Anal. Chim. Acta*, **11**, 18–27 (1954): *Chem. Abs.*, **49**, 6768 (25 May 1955).

Alkali-metal metaphosphates and polyphosphates dissolve Prussian Blue to $K_4Fe(CN)_6$ and to the corresponding alkali-metal ferric metaphosphates and polyphosphates; but smaller quantities peptise Prussian Blue. The alkali-metal salicylates and β-resorcylates also dissolve Prussian Blue if sufficient quantities are present, but smaller quantities yield colloidal Prussian Blue. It is incorrect to say that Turnbull's Blue is not peptised by $K_2C_2O_4$. It also seems that Turnbull's Blue is an equimolecular mixture of Prussian Blue and ferrous ferrocyanide. Data given in the literature describing some reactions leading to colloidal Prussian Blue and statements that Prussian Blue does not react with HBr are erroneous. C. O. C.

PATENTS

Aromatic Sulphonyl Halide Colour Couplers. Eastman Kodak Co. *USP* 2,678,882

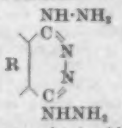
Compounds of formula—



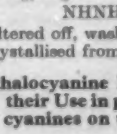
(R = group containing a complex group free to react with a primary aromatic amino silver halide developer; X = OH or Hal), e.g. α-(o-methoxybenzoyl)-4-[5-(2-carboxy-x-sulphobenzamido)-2-(2:4-di-*tert.*-amylphenoxy)benzamido]-2-methoxyacetanilide, are colour couplers readily incorporated into photographic emulsions as soluble salts. C. O. C.

1:4-Dihydrazinophthalazines. CFM. *BP* 732,521

Dye intermediates of formula—



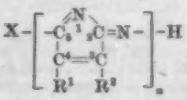
(R = heterocyclic ring) are obtained by treating o-dinitriles in which the nitrile groups are linked to a heterocyclic link with $\leqslant 3$ mol. of hydrazine in presence of a weak acid at $> 100°C$. Thus pyridine-2:3-dicarboxylic acid dinitrile (129 parts) and then 75% hydrazine hydrate (200) are added to dioxan (300). To the mixture is added during 1 hr. at 30°c. glacial acetic acid (40). The whole is stirred for 1 hr. at 90°c. and then heated for 3 hr. to 90–95°c. The product



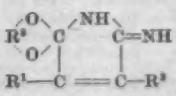
is filtered off, washed with dioxan and recrystallised from water. C. O. C.

Phthalocyanine Intermediates and their Use in producing Phthalocyanines on the Fibre. ICI. *BP* 730,467

Compounds of formula—



and



(X = NH_2, Alkoxy or Aralkoxy; R^1 and R^2 = H, Alk, Aralk or together form a *cyclo*hexene ring; R^3 = atoms completing a heterocyclic ring; $n > 0$) are used to produce

phthalocyanines on e.g. textile materials. They are prepared by condensing a maleic or fumaric dinitrile or a Δ^1-tetrahydrophthalonitrile with NH_3 or an alkyl-, aralkyl- or *cyclo*alkyl-alcohol in presence of an ancillary compound, viz. M–O–Y (M = alkali or alkaline earth metal or onium radical; Y = H or an organic radical). Thus NH_3 is passed through boiling methyl alcoholic ammonia containing Δ^1-tetrahydrophthalonitrile. After treating with charcoal and evaporating off the methanol, the residue is extracted with boiling ethyl acetate, from which the product, 2-imino-5-amino-3:4-tetrahydrobenz-isopyrrole separates on cooling.

BP 730,468

The above compounds are applied to e.g. textile materials with or without a metal-containing compound and a catalyst, e.g. ammonium molybdate; and a phthalocyanine formed *in situ* by subsequently heating the impregnated substrate. Thus, a printing paste containing 2-imino-5-amino-3:4-tetrahydrobenz-*iso*pyrrole, copper acetate, acetic acid and thickening, is printed on to cotton cloth which is dried and steamed (or preferably baked at 135–140°C.) for 10 min. The print is aftertreated in aq. formic acid at 90–100°C. and finally in soap solution.

R. K. F.

Leuco Metal Phthalocyanines. DuP. *USP* 2,681,347

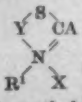
Reversible, solvent-soluble oxidation products of copper and nickel phthalocyanines are obtained by treating metal-free phthalocyanines with a halogen in an alcoholic solution containing a salt of copper or nickel. They are similar in properties to the leuco metal phthalocyanines obtained by the process of *USP* 2,662,896 and very likely have similar structures; they are dyed or printed on textiles by the methods described in that patent.

USP 2,681,348

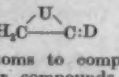
Phthalonitrile is treated with ammonia and CaO in an anhydrous solvent at 65–100°C. for 10–40 hr. using 1 mol. of CaO per 6 mol. phthalonitrile. A complex solvent-soluble calcium compound is obtained of empirical formula $Ca(C_8H_4N_2)_6{\cdot}NH_3{\cdot}3H_2O$. This complex has no tinctorial properties, its solution in alcohols, benzene or chloroform being a yellowish green. Upon reduction with aqueous and/or alcoholic solutions of ascorbic acid it decomposes to yield phthalonitrile, ammonia, calcium ascorbate and metal-free phthalocyanine. It can thus be used for producing phthalocyanines *in situ* in the fibre. C. O. C.

cyclo Ammonium Quaternary Salts—Intermediates for Polymethin Dyes. P. D. Dreyfus and B. Gaspar. *USP* 2,677,683

*cyclo*Ammonium quaternary salts of formula—



(A = subst. or unsubst. methyl; R^1 = Alk; X = anion; Y = chain of 2 or 3 C atoms) useful as intermediates for polymethin dyes are obtained by condensing 1 mol. of a compound of formula $R^2{\cdot}NH{\cdot}Y{\cdot}X$ with 1 mol. of a compound of formula $A{\cdot}CS{\cdot}N{\cdot}B_2$ (R^2 = Alk or Ar; B = H or its equivalents) preferably in presence of 1 mol. of an acid, HX, which serves to stabilise the compound $R^2{\cdot}NH{\cdot}Y{\cdot}X$ which otherwise tends to condense with itself. They condense with compounds of formula—

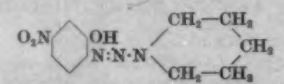


(D = O or S; U = atoms to complete a heterocyclic nucleus) or with other compounds having a reactive methylene group to yield merocyanines. Thus 2-methyl-3-phenylpenthiazolinium perchlorate melted at 130°C. with diphenylformamidine yields 2-(2-anilino vinyl)-3-phenyl-penthiazolinium perchlorate. This on being condensed with *N*-*n*-hexylrhodanine yields the dye 3-*n*-hexyl-5-[(3-phenyl-2(3)thiazolinylidene)ethylidene]rhodanine.

C. O. C.

Triazens for Production of Metallisable Azoic Dyes on Nylon. ICI. *BP* 730,653

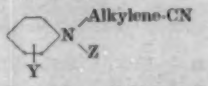
The triazens obtained by condensing diazotised *o*-aminophenols with amines NHR^1R^2 (R^1 = H, Alk, Aralkyl, or cycloalkyl; R^2 = Alk, aralkyl, or cycloalkyl, or R^1 and R^2 may be joined together to form with the N atom a heterocyclic ring) or with cyanamide, guanidine, or a subst. guanidine, are applied to nylon along with suitable coupling components, free of COOH or SO_3H groups, to give azoic dyes which may be metallised on the fibre with e.g. chromium or copper compounds. Thus diazotised 5-nitro-2-aminophenol is condensed with piperidine to give the triazene—



which is applied to nylon along with 2:7-dihydroxy-naphthalene at 95°C. from a salt bath. The azoic dye is then developed by treatment at 85°C. in dil. HCl. A dark brown is obtained by chroming on the fibre, or a maroon by treating with cuprous chloride and formic acid. E. S.

Monoazo Disperse Dyes for Cellulose Acetate, Nylon, etc. S. *BP* 731,448

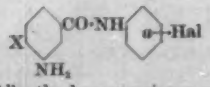
Yellow-brown to red disperse dyes are made by diazotising 2-bromo-4-nitroaniline, or its 6-bromo-, 6-alkyl-, or 6-SO_2·Alk derivatives, and coupling with *N*-cyanoalkyl derivatives of aniline—



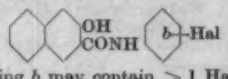
(Y = H or Alk; Z = Alk or hydroxyalkyl). Thus 2:6-dibromo-4-nitroaniline is diazotised in conc. H_2SO_4 and coupled with *N*-cyanoethyl-*N*-hydroxyethylaniline to give a yellow-brown dye for cellulose acetate, nylon and polyester fibres. E. S.

Monoazo Pigments for Plastics, etc. FH. *BP* 731,157

Amines—



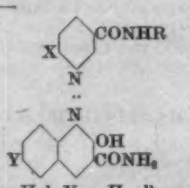
(X = Alk or OAlk; the benzene ring *a* may contain > 1 Hal atom) are diazotised and coupled with arylides of 3:2-hydroxynaphthoic acid.



(the benzene ring *b* may contain > 1 Hal atom) to give pigments which colour polyvinyl chloride resins, etc. red of good fastness to bleeding. An example is 3-amino-4-methoxybenz-2′:4′-dichloroanilide→3-hydroxy-2-naphtho-*p*-chloroanilide. E. S.

Monoazo Dyes for Colouring Polyvinyl Plastics. FH. *BP* 731,162

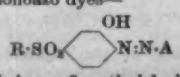
Dyes of formula—



(X = Alk, alkoxy or Hal; Y = H, alkoxy or Hal; R = H, hydroaromatic radical or subst. or unsubst. Alk, aralkyl or Ar), e.g. the bluish red dye 1-amino-2-methoxybenzene-5-carboylaminobenzene→2:3-hydroxynaphthoylamine, are useful for pigmenting plasticised polyvinyl compounds very fast reddish oranges to clarets. C. O. C.

Metal(Chromium)-complex Monoazo Dyes for Wool, Nylon, etc. Gy. *BP* 730,036

oo′-Dihydroxymonoazo dyes—

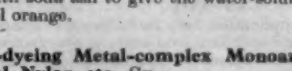


(R = Alk of 2–4 C; A = a 3-methyl-1-phenyl-5-pyrazolone or a 1-acylamino-7-naphthol with 1–3 C in the acyl residue) free from SO_3H or COOH, give water-soluble chromium

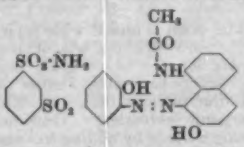
complexes which dye wool, nylon, silk and glacé leather from neutral or weakly acid baths. They exhaust more completely than dyes of *BP* 667,168 (R = CH_3). The diazo components may be made from *p*-chlorophenylsulphinic acid by alkylating with diethyl sulphate, propyl or butyl bromide, nitrating, replacing the Cl atom by OH using hot caustic alkali, and reducing the NO_2 group to NH_2. Thus the monoazo compound 2-aminophenol-4-*n*-propylsulphone →3-methyl-1-phenyl-5-pyrazolone is refluxed with aq. ammonium chromosalicylate. The complex so formed is mixed with soda ash to give the water-soluble dye which dyes wool orange. E. S.

Neutral-dyeing Metal-complex Monoazo Dyes for Wool, Nylon, etc. Gy. *BP* 731,114

oo'-Dihydroxy- and *o*-alkoxy-*o'*-hydroxy-monoazo dyes, free of SO_3H and COOH groups, containing a benzene sulphonamide group or groups attached through a bridging group, and of formula—



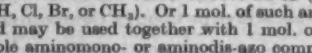
(X = *o*-OH or *o*-OAlk; A and Z = aryl of benzene series; B = radical of *ortho*-coupling coupling component; Y = CO, SO_2, or SO_2·NR, where R = Alk of 1–3 C; *n* = 1 or 2) are treated with metallic (especially Cr or Co) compounds, to give complexes which dye wool, nylon, etc., from neutral or weakly acid baths. They exhaust better from neutral dyebaths than similar dyes in which a sulphonamide group is directly attached to the chromophore system. Thus 2-aminophenol-4-phenylsulphone-3'-sulphonamide is diazotised and coupled with 1-acetylamino-7-naphthol to give—



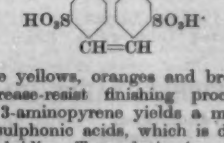
Boiling with aq. ammonium chromosalicylate gives the complex, which dyes wool grey. E. S.

Dis- and Poly-Azo Direct Cotton Dyes Containing a Pyrene Residue. S. *BP* 731,199

Dis- and poly-azo direct cotton dyes similar to those of *BP* 701,709 (*J.S.D.C.*, **70**, 163 (1954)), but containing a pyrene residue, are made by condensing 2 mol. of aminoazo compounds R^1·N:N·R^2-*w*-NH_2 (R^1 = aryl of benzene series; R^2 = radical of the pyrene series; *w* is a direct link or



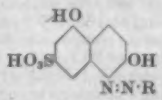
or a subst. derivative of these two radicals) with 1 mol. of a dicarboxylic acid halide X·CO·CA:CH·CO·X (X = Cl or Br; A = H, Cl, Br, or CH_3). Or 1 mol. of such an aminoazo compound may be used together with 1 mol. of any non-metallisable aminomono- or aminodis-azo compound with the exception of the aminoazo compound—



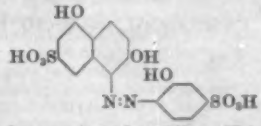
The products are yellows, oranges and browns of good resistance to crease-resist finishing processes. Thus, sulphonation of 3-aminopyrene yields a mixture of the 5:8- and 5:10-disulphonic acids, which is diazotised and coupled with *m*-toluidine. To a solution in aq. NaOH of the resulting aminoazo compound are added slowly a benzene solution of fumaric dichloride and sufficient soda ash to maintain a weakly alkaline reaction throughout. The product dyes cotton dischargeable reddish yellow. E. S.

Metal(Copper)-complex Polyazo Direct Cotton Dyes. S. *BP* 730,547

The copper complexes of polyazo dyes obtained by coupling tetrazotised dianisidine with 1 mol. of a monoazo compound (I)—



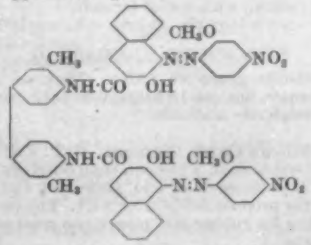
(R = the radical of a benzene or naphthalene series sulphonic acid and contains OH *ortho* to the azo link) and with 1 mol. of any other coupling component, dye cotton navy blue to grey blue. The monoazo compounds (I) may be prepared by coupling a diazotised aniline sulphonic acid with *O*-acetyl-J acid, exchanging the NH_2 group in the J acid residue with an OH group according to the process of *BP* 727,747, introducing an *ortho*-OH group into the benzene nucleus by oxidative coppering, and hydrolysing the *O*-acetyl group. Or, some of them may be prepared by coupling a diazotised *O*-acyl-J acid with a naphthol sulphonic acid, introducing a 2-OH group into the J acid residue by oxidative coupling as described in *BP* 660,447 (*J.S.D.C.*, **68**, 99 (1952)), and then hydrolysing the *O*-acyl group. The resulting copper complexes may be used for further coupling as such, or they may be de-coppered. Thus, tetrazotised dianisidine is coupled first with 1 mol. of 1-naphthol-3:6-disulphonic acid in presence of soda ash and then with 1 mol. of the copper complex of the monoazo dye—



in presence of ammonia and pyridine. This trisazo dye so formed is coppered by treatment with cuprammonium hydroxide in presence of pyridine which demethylates the dianisidine residue, and yields the navy blue direct cotton dye. E. S.

Azo Pigments. Ciba. *BP* 730,384

Insoluble azo compounds free of SO_3H and sulphonamide groups but containing a COOH group, and an OH group *ortho* to an azo group, are converted into acid halides, which are condensed with non-vattable cyclic amines to give pigments of good stability, suitable e.g. for pigment printing and for incorporation into plastics and spinning solutions of synthetic fibres. Thus the azo dye 5-nitro-2-aminoanisole→3:2-hydroxynaphthoic acid is treated with thionyl chloride in chlorobenzene, and the acid chloride so formed boiled in chlorobenzene with *o*-tolidine in the presence of pyridine to give—



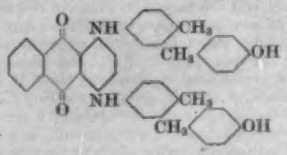
which, incorporated in polyvinyl chloride resins, gives a reddish violet, fast to migration. E. S.

Easily Destroyed Dyes for Use in Photographic Light-screening Layers. B. Gaspar, G. B. Linden, and P. D. Dreyfus. *USP* 2,677,612

Compounds of formula XYC:NZ (X = an organic radical; Y = H or an organic radical; X and Y are linked to the C atom by carbon to carbon bonds and together with the C atom may form part of a ring; Z = an organic residue), e.g. di(*o*-hydroxbenzene)-benzidine-2:2'-disulphonic acid, are relatively stable in alkaline and neutral baths but split in acid baths to form X·CO·Y and ZNH_2 which are colourless and/or readily washed out of the photographic colloid. C. O. C.

Disperse Dyes of Excellent Fastness to Dry Cleaning. General Aniline. *USP* 2,677,694

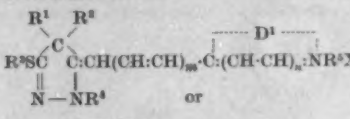
Disperse dyes of high tinctorial strength, brilliant hue, insoluble in water, hydrocarbons and chlorinated hydrocarbons but soluble in low mol.wt. alcohols and ketones are obtained by heating at 100–200°C. a chloromethylated arylaminoanthraquinone compound with a monocyclic phenol tar until evolution of HCl ceases. Neither compound should contain a water-solubilising group and at least one of the ortho and para positions to the phenolic hydroxy group must be unsubstituted. Thus the green dye—



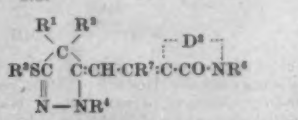
is produced by slowly heating 1:4-bis-(chloromethyl-*p*-toluidino)-anthraquinone (20 parts by wt.) with phenol (150) to 158°C., keeping at that temp. for 2 hr., diluting with 50% aqueous ethanol (300) to precipitate the dye, filtering, washing and drying. C. O. C.

Cyanine and *mero*Cyanine Dyes. Ilford. *BP* 730,489

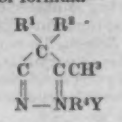
Dyes of formula—



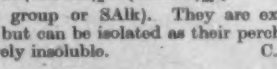
or



(R^1, R^2, R^3, and R^4 = lower Alk; R^5 = Alk; R^6 = hydrocarbon or is linked via C and N to a C atom in D^2 to form a further heterocyclic nucleus; R^7 = H or Alk; X = acid radical; m and n = 0 or 1; D^1 = atoms to complete a 5- or 6-membered ring; D^2 = atoms to complete a heterocyclic nitrogen keto methylene nucleus) are obtained e.g. by reacting a compound of formula—



(Y = acid radical) with one of formula—



(Q = acetanilido group or SAlk). They are extremely soluble in water, but can be isolated as their perchlorates, which are relatively insoluble. C. O. C.

Calcium-Silicon Oxide Pigment. DuP. *USP* 2,679,463

Aqueous solutions of a calcium compound and a silicate are mixed in presence of an acid, preferably carbonic acid, so that in the product SiO_2:CaO::>4:1. The products are used as fillers for rubber and plastic compositions, and in paper-coating. C. O. C.

White-Lead-containing Pigment. Eagle-Picher Co. *USP* 2,677,621

Pigment of low lead content, excellent colour, durability and hiding power is obtained by coprecipitating PbO and $MgCO_3$ from an aqueous slurry. C. O. C.

Colloidal Silica with an Organophilic Surface. FBy. *BP* 730,861

An aqueous silica sol. is treated with an aliphatic or *cyclo*aliphatic amine of > 6 C, e.g. octadecylamine or hexahydro-*o*-toluidine and the product filtered off and dried. The product is a filler for rubber, plastics, paints, inks, etc. C. O. C.

Dispersible Dye Powders. General Aniline. *USP* 2,678,256

Powders produced by grinding a stiff paste of a disperse dye and aqueous secondary liquorice extract until the dye is in colloidal form and drying the mixture to a dry powder, are readily dispersible in water and dye liquors made from them do not deposit insoluble tarry material even when boiled. C. O. C.

Carbon Black (I p. 469).
Oxidation Dyes (IX p. 481).
Analytical Application of Reflectancy Determination. I— Filter Paper Determination of Spectro-reflectancy of Coal Tar Colours for Foodstuffs. II—Determination of Surface Reflectancy of 2:4-Dinitrophenylhydrazones. III—Notation of Surface Colour of 2:4-Dinitrophenylhydrazones on C.I.E. Colour Chart. IV—Colours and Absorption Maxima of 2:4-Dinitrophenylhydrazones in Alkaline Solution (XIV p. 486).
Simple Rapid Method for determining the Dyeing Behaviour of Anionic Dyes on Chrome Leather (XIV p. 488).

V—PAINTS; ENAMELS; INKS

PATENTS

Water Dispersion Paint. Firestone Tire & Rubber Co. *BP* 731,091

A pigment is dispersed in a latex containing a conjugated diene (< 50 parts by wt.) and an ethylenic compound (> 50), e.g. butadiene-1:3 copolymerised with styrene. C. O. C.

Press-stable, Moisture-sensitive, Shellac-containing Printing Inks. A. F. Schmutzler. *USP* 2,678,278

Fluorescent Compositions. John Waddington. *BP* 730,555

A soluble alkyd resin is mixed with (*a*) a suitable basic dye (0·05–2·0% pure dye on wt. of resin), (*b*) silica, alumina, complex silicate or complex aluminate, (*c*) as solvent a ketone and/or ester and/or ether and/or aromatic hydrocarbon and/or nitroparaffin, and, if desired, (*d*) an inorganic or organic extender. Thus an ink suitable for screen printing is made by adding to Paralac 385 (1 kg.) at 160°C., Rhodamine BN 450 (C.I.749) (6 g.), cooling to 120°C. and then adding ethyleneglycolmonomethylether (700 c.c.) and Standard Neosyl (700 g.). It has good fastness to light and weathering. C. O. C.

Coating Composition containing a Water Soluble Fluorescent Dye. H. Grossman. *USP* 2,681,317

Aqueous dispersions of a drying oil, resin, e.g. dewaxed shellac, filler or pigment, e.g. aluminium stearate or barium carbonate, are mixed with an aqueous solution of a fluorescent dye, e.g. Rhodamine B. The mixture is then coagulated by adding an electrolyte, e.g. HCl. This transfers the dispersed materials from the water to the oil phase. The oil phase is then filtered from the released water and dried. It is ready for use and can be thinned by adding a drying oil or an oil solvent, e.g. turpentine. C. O. C.

Polytetrafluoroethylene Coating Compositions. DuP. *USP* 2,681,324

Coating compositions which mostly yield relative thick films in one application without any tendency to crack during drying and/or fusing are obtained by adding to an aqueous dispersion of polytetrafluoroethylene a solid modifying polymer and/or solvent-swollen particles and/or an organic solution dispersed in a liquid mainly composed of water. C. O. C.

Fire-retardant Coating Composition (III p. 471).
Clear, Pigmented Oil-in-water Resin Emulsions (IX p. 481).

VI—FIBRES; YARNS; FABRICS

Existence of Individual Fibrils in Native Fibre Cellulose. H. Dolmetsch. *Melliand Textilber.*, **36**, 419–425 (May 1955).

It is possible to weaken the interfibrillary cement in native fibres, e.g. cotton or holocellulose, without attack of the cellulose by a treatment with hot hydrazine hydrate solution. By this means a breaking down of the fine structure can be carried out in a controlled manner to

such a degree that the crystalline regions are not attacked. This proves the existence of individual fibrils of such an order of magnitude that they can be observed, photographed, and measured under a visual microscope, obviating previous sources of error. S. R. C.

Formation of Carbonyl Sulphide in Viscose Manufacture. B. Philipp. *Faserforsch. und Textiltech.*, **6**, 13–19 (Jan. 1955).

A polarographic method for the determination of COS (or the equiv. monothiocarbonates) in presence of large amounts of CS_2 is applied in a study of the variation of COS concn. throughout the viscose process. Whereas in the reaction of NaOH soln. with CS_2 the COS concn. rises rapidly to a max. and then falls owing to the occurrence of further reactions, in the xanthation of alkali-cellulose it rises to 0·1–0·4% by wt. in about 2 hr. and then remains const., the COS being largely preserved from further reaction by combination with cellulose. During ripening the COS concn. falls rapidly to a min., rises again to a max., and then falls again, and it is shown that this behaviour is in accord with generally accepted views on the course of the process. A. E. S.

Heat of Wetting of Silk Fibroin by Water. H. B. Dunford and J. L. Morrison. *Canadian J. Chem.*, **33**, 904–912 (May 1955).

Heats of wetting by water of silk fibroin containing varying amounts of adsorbed and desorbed water have been measured. These measurements, with a water vapour adsorption isotherm, are used to calculate thermodynamic quantities. In contrast to cellulose, silk containing desorbed water evolves less heat than that containing adsorbed water, suggesting that any contribution by a heat of swelling term is very small and that the calculated quantities can be assigned almost entirely to adsorption. Changes in heats and entropies of adsorption seem to parallel the sequence of changes in film formation. W. R. M.

Yellowing of Silk. II—Chemical Mechanism. J. Pinte, Y. Teyssier, and P. Rochas. *Bull. Inst. Text. France*, (52), 17–39 (April 1955).

Yellowing of silk is accompanied by peptide chain breakdown and diminution of the orientation of the fibre, as shown respectively by increase in fluidity and decrease in swelling anisotropy. Yellowing also produces an increase in alkali sorption capacity which counteracts the decrease in this factor produced by ageing under conditions which do not induce yellowing. No difference was found in total N content and amino acid composition of a reference sample and samples aged with and without yellowing. The yellow colour could be extracted with water. The extract contained no free amino acids, but after hydrolysis glycine, alanine, and serine were present, together with a small amount of aspartic acid and a trace of tyrosine. A mechanism of oxidative fission, or of hydrolytic fission followed by oxidation, according to the conditions under which yellowing takes place, is suggested. J. C. F.

Formation of Fibre from Groundnut Protein. W. E. F. Naismith and R. H. K. Thomson. *J. Appl. Chem.*, **5**, 192–196 (April 1955).

Groundnut protein, in the form of a suitably prepared soln., is examined in the ultracentrifuge at each stage of fibre manufacture. Also, a series of variants of the spinning soln. (aged in the same way, but varying in concn. of protein and NaOH) is examined rheologically, and the results are correlated with ultracentrifuge measurements on the same soln. after dialysis. Little change occurs in the protein during the alkaline-extraction and acid-reprecipitation processes, but the preparation of a usable spinning soln. results in the disappearance of the three characteristic sedimentation peaks corresponding to the three individual proteins originally present (depending on the conditions of preparation, the soln. may no longer contain material of very high mol.wt., or such material may be present in a polydisperse form). After extrusion and coagulation, only very slowly sedimenting material is present. A. E. S.

Macromolecular Compounds. LXIX—Dependence of the Properties of Polyamides on the Extent of Hydrogen Bonding. LXXIII—Some Two-component Mixed Polyamides. V. V. Korshak and T. M. Frunze. *Izvestiya Akad. Nauk S.S.S.R., Otdel. khim. nauk*, 163–171 (Jan.–Feb.); 372–379 (March–April 1955).

LXIX—Mixed polyamides are prepared from a mixture of the salts formed by hexamethylenediamine with two of the following dicarboxylic acids—succinic, adipic, suberic, azelaic, and sebacic. Their m.p. and solubilities are determined, and these and other data form the basis of a discussion of the extent to which hydrogen bonding can occur in simple and mixed polyamides (cf. J.S.D.C., **71**, 114 (Feb. 1955)).

LXXIII—The investigation is extended to include 6-hexanolactam as one component of the reaction mixture, the other being hexamethylenediamine adipate, azela-ate, or sebacate. In each binary series a minimum m.p. is found at a mole fraction of 0·8 of 6-hexanolactam. A. E. S.

Effect of Orientation on the Sorptive Power of Crystalline Polymers. V. A. Kargin and T. V. Gatovskaya. *Doklady Akad. Nauk S.S.S.R.*, **100**, 105–106 (1 Jan. 1955).

The adsorption of vapours at low pressure on polyethylene, 6-nylon, and 6,6-nylon in the undrawn and cold-drawn (500% draft for polyethylene and 6-nylon and 800% for 6,6-nylon) states is measured with the aid of a McBain sorption balance, the sorbates being pentane and formic acid for 6-nylon and hexane for polyethylene and 6,6-nylon. Sorption by 6- and 6,6-nylons is increased by drawing, an observation that is in conflict with the view that drawing is accompanied by increase in the density of packing of the macromolecules. On the other hand, sorption by polyethylene is decreased by drawing, but this behaviour cannot be interpreted unequivocally as the result of increased density of packing, since the reduced flexibility of the oriented molecules can itself lead to reduced adsorption. A. E. S.

PATENTS

Modified Viscose Yarns. Courtaulds. *BP* 730,541

Yarns having substantially no skin and core effect are produced by extruding viscose into a coagulating bath containing H_2SO_4, $ZnSO_4$ and 0·25–12 millimoles of fluorescein per 100 g. of viscose. W. G. C.

Casein Fibres. American Patents Corporation. *BP* 728,749

Fatty compounds may be permanently fixed in regenerated protein fibres by adding to the spinning solution an emulsion of $< 5\%$ of a highly unsaturated triglyceride fatty oil, e.g. bleached herring oil. W. G. C.

Linear Polyamides from Pyrrolidone. Arnold, Hoffman & Co. *BP* 731,294

The polymers of formula—

$$-\left(-NH\cdot CH_2\cdot CH_2\cdot CH_2\cdot CO-\right)_x-$$

obtained by heating a pyrrolidone in presence of an alkali metal or its oxide are thermoplastic fibre-forming solids of m.p. 250–260°C., soluble in conc. HCl from which they can be precipitated by adding a base. C. O. C.

Acrylonitrile Copolymers. Celanese Corpn. of America. *BP* 729,002

Copolymers of high intrinsic viscosity and good solubility are obtained by treating acrylonitrile with an amide of an $\alpha\beta$-unsaturated monocarboxylic acid, e.g. *N-cyclo*hexyl acrylamide, in an aqueous polymerisation medium containing enough tertiary butyl alcohol to dissolve the monomers. Filaments formed from them have good affinity for dyes. W. G. C.

Acrylonitrile Copolymer Solution. Celanese Corpn. of America. *BP* 730,513

Solutions of copolymers of acrylonitrile with an amide of $\alpha\beta$-unsaturated monocarboxylic acid, e.g. *N-cyclo*hexyl acrylamide in *cyclo*pentanone, *cyclo*hexanone and nitromethane are suitable for spinning into fibres. W. G. C.

Removal of Porosity in Wet Spun Acrylonitrile Filaments by Treatment with Hot Fluid or with a Hot Surface. *USP* 2,677,590–1

Acetone-soluble Acrylonitrile-Vinylidene Chloride Copolymers. Dow Chemical Co. *USP* 2,681,325

Copolymers of 30–55% by wt. of acrylonitrile and 70–45% of vinylidene chloride readily yield 10–20% solutions in acetone. Such solutions can be used for the dry but are preferably used for the wet spinning of fibres.

C. O. C.

Improving the Dyeing Properties of Polyacrylonitrile. Distillers Co. *BP* 731,056

Incorporation of > 10% of a fusible, soluble and uncured phenol-aldehyde condensate into acrylonitrile polymers and copolymers much improves their affinity for disperse dyes.

C. O. C.

Solutions of Acrylonitrile Polymers or Copolymers (III p. 471).

Sizing Glass Fibres (X p. 482).

Reducing the Plastic Extensibility of Synthetic Linear Polymer Textiles (X p. 484).

Granules coated with Pigment (XIII p. 485).

Determination of Sulphide Sulphur and Reducible Sulphur in Viscose Rayon Materials (XIV p. 487).

Ion-exchange Separations on Chemically Modified Cellulose (XIV p. 488).

VII— DESIZING; SCOURING; CARBONISING; BLEACHING

Desizing Procedures in relation to Enzyme Evaluation. H. C. Borghetty and J. T. Taylor. *Amer. Dyestuff Rep.*, **44**, P 256–P 261 (11 April 1955).

Use of heat-stable enzymes for high-speed desizing makes accurate evaluation of enzyme preparations increasingly important. It is considered that for textile purposes the liquefaction method is most suitable, as the end-products must be removed as easily as possible. Brief data are given on methods of high-speed desizing, and a detailed description is presented of the Rohm & Haas enzyme evaluation method, and of a brief procedure suitable for laboratory comparison. J. W. B.

Action of Chlorine-liberating Detergents on Crease-resist Finishes. W. Graeser. *Melliand Textilber.*, **36**, 459–462 (May 1955).

Amino resins absorb chlorine, and in storage and under illumination the complex results in fibre damage. Since the chlorine is bound by free NH_2 groups, it was believed that the greater the uptake of halogen the greater the number of such groups, i.e. the lower the condensation temperature, but this is complicated by the fact that these alkaline media hydrolyse the resin. The chlorine uptake can be used on the goods as a measure of resin stability. Fade-Ometer illumination breaks down the finish, which is reflected in higher chlorine uptake. A normal soap-and-soda wash reduces the chlorine uptake in parallel with the reduction of resin content. Finality in resinification is not reached by condensation in saturated steam. In absence of catalyst, autoresinification takes place, by the formation of proton bridges between amino and hydroxymethyl groups, after previous splitting-off of formaldehyde.

S. R. C.

PATENTS

Bleaching Cellulosic Material. ICI. *BP* 729,749

Treating first with a hot aqueous solution of a chlorite, then with alkali and again with hot aqueous chlorite gives as good a bleach as with the use of a single chlorite treatment with steaming and uses less chlorite. C. O. C.

Bleaching with Hydrogen Peroxide. DuP. *USP* 2,679,483

An improved sodium silicate solution for use in hydrogen peroxide bleaching baths is obtained by mixed sodium silicate and NaOH while limiting the amount of water present so that a temperature of > 60°c. is obtained. The resulting solution is then diluted when required for use.

C. O. C.

Purifying Raw Wool by Anaerobic Fermentation. Pacific Flush Tank Co. *BP* 730,037

Raw wool is introduced without prior treatment into a liquor seeded with active microorganisms capable of anaerobically fermenting organic components of adhering material, i.e. wool grease and cellulosic burrs, without damaging the wool. The liquor may be prepared with a bacterial culture or may be from an active sewage sludge digestor tank. Fermentation is commenced by adding a small amount of wool which is gradually built up as the reaction progresses; it is maintained by controlling volatile acid and ammonia. Approx. 60% of the vegetable matter is converted to gas and a reduced carbonising treatment suffices at a later stage. After draining, centrifuging and rinsing no scouring is necessary. J. W. B.

Alkaline Peroxide Bleaching of Wool. DuP. *USP* 2,676,871

Wool may be rapidly bleached with hot alkaline peroxide without excessive peroxide consumption and with minimum damage to the wool, if the wool is first treated so that it contains 0·5–1·0% of combined HCHO.

C. O. C.

Laundering Textile Materials. E. Sulzmann. *BP* 730,790

Economies in liquor and materials are effected in the laundering of household textiles, e.g. hospital linen which is not very soiled, by using a number of washing machines arranged in a ring so that liquor can circulate throughout or can be cut off from individual machines or sections while the different stages are being carried out. J. W. B.

VIII— DYEING

Historical Notes on the Wet-processing Industry. VIII— The Secrets of the Reverende Maister Alexis of Piemount. S. M. Edelstein. *Amer. Dyestuff Rep.*, **44**, 247, 270–272 (11 April 1955).

Pad–Roll Dyeing Process and its Technical and Economic Utilisation. C. O. Eriksson, N. O. Landqvist, and B. F. Mellbin. *Textil-Rund.*, **10**, 51–64 (Feb. 1955).

A review of a process in which fabric impregnated with dye liquor is wound on a cylinder, which is rotated for such a time and at such a temperature that uniform penetration of dye results. A discussion which followed the presentation of the paper is reported. J. C. F.

Gas Fume Fault on Wool. D. Holgate. J.S.D.C., **71**, 183–184 (April 1955).

Both moisture and light appear to play a big part in the fading (cf. J.S.D.C., **71**, 46 (Jan. 1955)). Residual sulphamic acid seems to confer resistance to the attack of burnt gas fumes. C. J. W. H.

Nylon Dyeing. A. E. Stubbs; A. B. Pakshver. J.S.D.C., **71**, 182–183 (April 1955).

Attention is drawn by Stubbs to errors in a series of papers by Pakshver and his co-workers (see J.S.D.C., **70**, 520 (1954)). In a reply, Pakshver accepts certain of Stubbs's criticisms, but insists that the attempt to express fibre structure by means of diffusion coefficients calculated mathematically is new. C. J. W. H.

Carriers in Dyeing Dacron Polyester Fibre. C. L. Zimmerman, J. M. Mecco, and A. J. Carlino. *Amer. Dyestuff Rep.*, **44**, P 296–P 302 (25 April 1955).

The carrier action with regard to disperse dyes of methyl salicylate (8 g./litre) is compared with that of phenylmethylcarbinol (20 g./litre) and *o*-phenylphenol (3 g./litre) and monochlorobenzene (4 g./litre). It is shown that methyl salicylate, emulsified e.g. with dioctyl sodium sulphosuccinate, gives very greatly improved results, complete fibre penetration usually being obtained in 20 min. or less at 180°F. Complete recipes are given.

J. W. B.

Piece Dyeing and Finishing of Polyacrylonitrile Goods— II. J. Müller. *Melliand Textilber.*, **36**, 470–476 (May 1955).

Process 1F (CFM) is considered and compared with Pattern Card F 3005 (DH). Blues obtained with Anthrasol Blue IBC (FH) and Alcian Blue 8 GX (ICI) are described. The dyeing of vats on PAN fibres, the dyeing of the same

fibres in admixture with cotton or viscose rayon staple, antistatic finishing, heat fixation, and the finishing of PAN marquisettes and gaberdines are referred to.

S. R. C.

PATENTS

Dyeing Cotton or Regenerated Cellulose with Inorganic Metallic Compounds. Crosland & Pickstone. *BP* 730,601

When cotton is impregnated with a ferrous or ferric salt and then, preferably after drying, treated with caustic lye and then the whole sequence of operations repeated, the resulting dyeing is redder and is more than twice as deep as that obtained with a single treatment. If the lye used is of mercerising strength the dyeing is deepened still more.

C. O. C.

Improving the Fastness of Colouring Matters in Textiles and Paper. Bradford Dyers' Assocn. *BP* 729,643

Dyes and pigments can be fixed fast to wet processing in textiles or paper and in absence of a filler by treating the material during or after application of the dyes or pigments with compounds of formula $R'(N{:}CX)_n$ or $R'(NH{\cdot}CXR^1)_n$ (R' = an organic residue which when it is aliphatic and $n = 1$ is of < 8 C; R^1 = residue of an organic compound containing one or more H atoms replaceable by alkali metals; X = O or S, $n = 1$, 2 or 3), e.g. hexamethyldiisocyanate or the adduct of 1 mol. of hexamethylene diisocyanate with 2 mol. of diethyl malonate, and then baking, e.g. at 100–200°C. for 15–3 min.

BP 729,656

Adducts of compounds of formula $R'(N{\cdot}CX)_n$ with alkali metal bisulphites are similarly used. C. O. C.

Dyeing Mixtures of Animal Fibres and Nylon—Reserving Agents for Nylon. S. *BP* 731,005

Nitrate, bromide, iodide or thiocyanate ions exert a strongly specific reserving effect on nylon against acid and afterchrome dyes. C. O. C.

Dyeing and Finishing Hosiery. Smith, Drum & Co. *USP* 2,678,868

Nylon or other thermoplastic stockings are preboarded, removed from the forms and stacked flat upon one another to form a bundle in which the preboarded state is maintained. They are held together as a stationary stack by means of a pervious cover or other device. The flat stack of stockings has dye and finishing liquors circulated through it. They are then dried by air being passed through the stack after which the stockings are ready for inspecting, pairing and boxing. C. O. C.

Dyeing Lignocellulose Hardboards. Masonite Corpn. *USP* 2,678,588

Hydrolysed lignocellulose fibres are formed into a sheet from an aqueous slurry. The sheet is passed between pressure rollers and the topside sprayed with a dye. It is then put on a screen sprayed side up and consolidated by heat and pressure. This results in a product whose smooth side is uniformly dyed to a considerable depth whereas the other side is less deeply dyed, the interior of the board being undyed. C. O. C.

Rendering Dyed Brillantine Fast to Light. Colgate-Palmolive Co. *USP* 2,678,901

Addition of benzoyl resorcinol to dyed brillantine imparts fastness to light. C. O. C.

Protecting and Colouring of Aluminium and Aluminium Alloys. United Anodising. *BP* 731,247

The metal is first surface oxidised, and then dipped in a solution containing radicals which form no precipitate in the bath but which when dried on the metal form a coloured inorganic compound in the oxide coating and seal it. Thus anodised aluminium immersed in a strong solution of nickel dichromate at 80°C. and pH 5–6 for 15 min. is dyed yellow fast to hot water. C. O. C.

Black Oxide Films on Aluminium. G. L. Deniston. *USP* 2,681,873

A black coating is given to aluminium by immersing it in a 10–17% aqueous solution of the following mixture — sodium chromate (26·0–30·6 parts by wt.), sulphuric acid (9·4–22·4), hydrochloric acid (14·8–34·6), potassium permanganate (26·5–30·6) and alkylarylsulphonate (1·2–2·2). C. O. C.

Antistatic Finish for Synthetic Polymeric Fibres—Increasing the Depth of Dyeings with Acid and Mordant Dyes (X p. 483).

Adsorption of Brazilwood and Logwood Colouring Matters by Fibres (XIV p. 488).

Review of Several Methods of Hydrosulphite Analysis with special regard to Control of Vat Dyebaths (XIV p. 488).

Simple Rapid Method for determining the Dyeing Behaviour of Anionic Dyes on Chrome Leather (XIV p. 488).

IX—PRINTING

Use of Thickenings in Vat Printing. W. Jülicher. *Melliand Textilber.*, **36**, 477–483 (May 1955).

The Colloresin process and the use of the Alkaprint process based on an ether of locust-bean gum are examined.

S. R. C.

Three-dimensional Coloured Styles on Cotton produced with Resins. E. W. Lawrence and R. H. Phillips. *Amer. Dyestuff Rep.*, **44**, P 253–P 255 (11 April 1955).

Methods are outlined for producing raised colour prints on cotton—either by impregnating with thermosetting resins, partly drying, emboss-calendering with inked cylinder, and curing; or by adding pigments to a resin printing mixture which contains a thermosetting resin which will resist the shrinking action of NaOH, and then passing through a caustic soda bath. J. W. B.

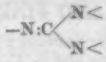
Printing and Related Operations on Glass Decorative Fabrics. R. F. Caroselli. *Amer. Dyestuff Rep.*, **44**, P 303–P 304 (25 April 1955).

A brief description is given of glass printing, comprising coronising to burn off finishes and relax the fibres, giving a protective coating of polyacrylate–latex containing dispersed Teflon, printing with resin pigments by both the water and oil systems, curing, and soaping. J. W. B.

PATENTS

Vat Dye Printing Pastes. CFM. *BP* 731,763

Addition to vat dye printing pastes of condensates of aldehydes with compounds containing at least one



group, e.g. HCHO with dicyandiamide, stabilises the prints so much that the final printing result is largely independent of the time that elapses between drying and steaming.

C. O. C.

Oxidation Dyes. Fran. *BP* 730,106

The mono-*N*-sulphonates of benzene diamines are printed on to materials consisting of animal, vegetable or superpolyamide fibres, in presence of an oxidising agent, e.g. a chlorate, and a catalyst, e.g. a vanadate, and heated under acidic conditions to produce browns. Thus a cotton fabric is printed with a paste containing sodium *p*-phenylenediamine-*N*-sulphonate, $NaClO_3$, NH_4Cl, ammonium vanadate, ammonia and a thickener. After drying, the material is neutral steamed (decomposition of the NH_4Cl produces the necessary acid conditions).

R. K. F.

Clear, Pigmented Oil-in-water Resin Emulsions. L. Auer. *USP* 2,681,322

A binder for coloured emulsions is obtained by using fatty acid ester resin binders which are emulsion polymerised or aggregated, e.g. alkyd resins, condensation products of bis-phenol and epichlorohydrin esterified with fatty acid esters, styrenated alkyds, styrenated oils or pentaerythritol esters of fatty acids. The esters are bodied to a suitable viscosity, emulsified and then treated with oxygen, e.g. by use of H_2O_2 or by blowing air through at 50–80°C. and pH < 5·7. The product is then mixed with a dispersion of a pigment and other necessary ingredients to yield a clear coloured product which may be used for obtaining textile prints of good fastness to rubbing, dry cleaning and washing, or for coating wood.

C. O. C.

Colour Photography. Gevaert. *BP* 731,268

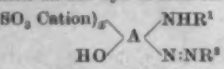
A multi-colour non-reversal image is formed by exposing to light a photographic multi-layer material containing colour couplers in the layers, colour-developing in a solution containing no colour coupler and re-exposing to light of a given colour while this colour-development is in progress. C. O. C.

Colour and Contrast Marking in Mono- or Polychrome Photography. F. H. Smith and D. O. Nicoll. *BP* 730,376

Preventing Colour Fog or Stain in Colour Photography. Kodak. *BP* 731,301

Azo Dye Bleachout Image. B. Gaspar and P. D. Dreyfus. *USP* 2,681,856

A method of producing three primary colours in the three layers of a photographic material using as a coloured coupling component an azo dye of formula—



(A = naphthalene residue; R^1 = H or hydrocarbon; R^2 = residue of a diazo compound; x = 1 or 2; the $N:NR^2$ group is *ortho* to the NHR^1 group and a function *ortho* to the OH group must be free to couple with a diazo compound), e.g. (2 : 5-dichlorobenzene)-(1-azo-7)-(8-amino-1-naphthol-3 : 6-disulphonic acid). In addition two colourless coupling components are used. The dye is in the red sensitive layer and is converted into a blue dye by the action of a diazo compound, the colourless couplers in the green and blue sensitive layer being converted to magenta and yellow dyes respectively in the same manner. C. O. C.

Printing on Blown Plastic Bottles having Flexible Walls. Peerless Roll Leaf Co. *BP* 731,649

Heat of Oxidation of Sodium Formaldehyde-sulphoxylate (III p. 470).
Phthalocyanine Intermediates and their Use in producing Phthalocyanines on the Fibre (IV p. 475).
Easily Destroyed Dyes for Use in Photographic Light-screening Layers (IV p. 477).
Improving the Fastness of Colouring Matters in Textiles and Paper (VIII p. 481).

X—SIZING AND FINISHING

Some Aspects of the Mechanical Finishing of Cotton Textiles. G. M. Nabar and G. P. Tawde. J.S.D.C., **71**, 343–350 (June 1955).

Some aspects of mechanical finishing processes for cotton textiles, mainly calendering, have been examined, and the influences of heat, pressure, moisture content, and starch content on the resultant finish investigated.

An increase in moisture content of the fabric during calendering produces the following effects on the finished samples—increase in stiffness and lustre, decrease in thickness, loss in textile qualities, and increase in resistance to abrasion. On storage, both the stiffness and the lustre of the calendered fabric show a gradual fall. With softening agents, the stage at which they are added to the starch paste determines the viscosity of the paste and the stiffness of the fabric. Studies on soluble starches show that for pastes of the same viscosity the stiffness of the fabric depends on the amount of the starch taken up and on the concentration of the starch. AUTHORS

Factors involved in Cropping. J. Cegarra Sanchez. *Teintex*, **20**, 345–357 (May 1955).

Each cloth has its appropriate shearing factor, which may be deduced from a single formula, being directly proportional to the density of fibres to be cut and inversely proportional to the cloth speed. The intensity of shearing increases with the diameter and speed of the blade roll. A 16-blade hollow bed appears to be more effective than a 10-blade hard bed, and stability to vibration of the former is better. S. R. C.

Resin Finishing of Textiles. P. Egli. *Textil-Rund.*, **10**, 64–69 (Feb. 1955).

A review of methods of imparting crease-resistance to textiles, and the evaluation and identification of such finishes. 44 references. J. C. F.

Urea–Formaldehyde Condensation. X—Presence of Dimethylene-ether Bridges in Condensates. G. Zigeuner and R. Pitter. *Mh. Chem.*, **86**, 57–68 (Feb. 1955).

Evidence is presented to show that the products of self-reaction of mono- and of bis-hydroxymethylurea and of reaction between urea and formaldehyde (all in alkaline media) are biscarbamidomethyl ethers and not hydroxymethylmethylene cpd.

XI—Methyleneureas. G. Zigeuner, R. Pitter, H. Berger, and H. Rauch. *Ibid.*, 165–172.

Urea and formaldehyde interact in acid soln. to form methylenediurea, which then reacts further forming linear methyleneureas—

$$H_2N{\cdot}CO{\cdot}NH{\cdot}[CH_2{\cdot}NH{\cdot}CO{\cdot}NH]_n{\cdot}CH_2{\cdot}NH{\cdot}CO{\cdot}NH_2$$

in which n has values of up to 6. The mechanism of this reaction is discussed.

XII—Hydroxybenzylcarbamides. G. Zigeuner, R. Pitter, and H. Rauch. *Ibid.*, 173–181.

The splitting of condensates by means of *m*-4-xylenol and identification of the products allows valuable conclusions to be reached about the structure of the condensates, but only if carried out under certain conditions (alcoholic HCl). H. E. N.

Acrolein–Formaldehyde—New Chemical for Rayon Stabilisation. P. B. Stam. *Amer. Dyestuff Rep.*, **44**, P 251–P 252, P 261 (11 April 1955).

Acrolein reacts with a mixture of formaldehyde and paraformaldehyde in presence of soda ash as catalyst and, if desired, in presence of small amounts of gums and starches for modification of the final product to give what has hitherto been known as "X2". Studies have covered a range of molecular ratios acrolein : formaldehyde from 1 : 1 to 1 : 6, and indicate that a ratio of 1 : 4 (part of the 4 can be paraformaldehyde) is best, with 1% of Na_2CO_3 as catalyst. The product is ready for use after neutralising with a mineral acid. It is considered that the compound may be very useful in rayon stabilisation and in crease-resistance of cotton; it is free from Cl retention, though, for viscose rayon, lacking in bouncy resilience and in dye-fixation for direct dyes. J. W. B.

Nitrile Latices—A New Concept in Textile Finishing. N. H. Sherwood. *Amer. Dyestuff Rep.*, **44**, P 262–P 264 (11 April 1955).

The nitrile latex Hycar (Goodrich) provides an excellent warp size for blue denim, the fabric having a high permanent abrasion resistance and increased fastness to laundering, as the indigo pigment is firmly bound. Fabrics with less warp on the face may be given increased abrasion resistance by padding through dilute latex baths. The polar nature of the nitrile particles enhances the adhesion to textile fibres, a property which may be utilised in binding pigments or loading materials which must withstand laundering. Mill trials are described and discussed. J. W. B.

PATENTS

Sizing Glass Fibres. Owens-Corning Fiberglas Corpn. *BP* 729,754

The abrasive effect of glass upon glass is reduced by protecting the glass filaments from each other with a microscopic discrete cushioning means, e.g. the filaments are coated with a slurry of very short cellulosic fibres. W. G. C.

Flexible Glass Filaments. B. F. Goodrich Co. *BP* 731,812

Coating glass filaments with metal greatly increases their resistance to flexing, e.g. thirty-six 0·000351 in. diameter glass filaments coated with a 0·0000348 in. layer of stainless steel were twisted into a yarn which when flexed through 150° at a constant rate had 3800 times the life span of a similar yarn whose filaments had not been coated. C. O. C.

Improving the Suppleness and Receptivity for Dyes of Glass Fibre Yarns or Fabrics. Société Anonyme des Manufactures des Glaces et Produits Chimiques de Saint-Gobain, Chauny et Cirey. *BP* 730,713

The material is heated to relax all stresses, then immediately and suddenly cooled and coated with a mixture of one or more thermosetting condensation resins and one or

more cationic active substances, e.g. glass fibre yarn is heated to 550°c. for 10 min., suddenly cooled and treated for 15 min. in a bath containing 2% of a urea–formaldehyde precondensate, 1% of Fixogene NT (Fran), 1% emulsified paraffin wax, 1% of a dicyandiamidine–formaldehyde condensate and 3% of formic acid. It is then dried at 80°c. and baked at 140°c. for 8 minutes. C. O. C.

Sizing Glass Yarn. Mo och Domsjo. *BP* 730,228

On emerging from the spinning nozzle glass filaments are coated with an aqueous solution of a water-soluble cellulose derivative, e.g. ethylhydroxyethyl cellulose, or an alginate (preferably 0·5–3·0% by wt.), a surface tension reducing agent (0·01–1·0%), and if desired a mineral or vegetable oil (e.g. 0·3% in emulsion). This improves the lubrication and general filament properties. J. W. B.

Treating Regenerated Cellulose with Aldehydes. American Viscose Corpn. *USP* 2,679,449

The dry material is treated in a hot aqueous bath containing formaldehyde, glyoxal or pyruvic aldehyde, an acid catalyst, e.g. HCl, and a water-miscible liquid which is neither a non-swelling agent for the fibre nor reactive with the aldehyde at acid pH. The temperature of the bath is such that reaction between the fibre and the aldehyde is completed before the material is taken out. Very uniform readily controlled treatment is obtained. C. O. C.

Durable Antistatic Finish. Monsanto. *USP* 2,676,896

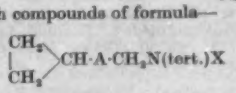
Treating with a water-insoluble amine salt of sulphonated polystyrene imparts a soft, antistatic finish of good fastness to washing and dry cleaning. C. O. C.

Antistatic Finish for Synthetic Polymeric Fibres—Increasing the Depth of Dyeings with Acid and Mordant Dyes. Monsanto. *BP* 730,210

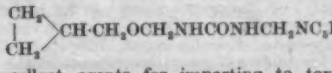
Textiles containing $< 10\%$ of synthetic polymeric fibres are given an antistatic finish by treatment with a quaternary phosphonium salt, e.g. nylon is impregnated with 80% of its wt. of a 2% soln. of benzyl tris(dimethylamino)-phosphonium chloride. The phosphonium salts may be applied in an acid dyebath when in addition to imparting an antistatic finish they also result in deeper dyeings. C. O. C.

Combined Water-repellency, Dimensional-stability and Crease-resistance. Deering Milliken Research Trust. *USP* 2,680,057

Treatment with compounds of formula—



(A = bi- or trivalent aliphatic, *cyclo*aliphatic or aromatic radical and may contain an ether, imino, carboxyl, carboxylic amide, carboxylic ester, subst. urea or subst. carbamate linkage; X = anion; N(tert.) = residue of a *tert.* amine), e.g. the pyridinium salt of *cyclo*propyl methyl ether of dimethylol urea,



are excellent agents for imparting to textiles water-repellency combined with dimensional stability and crease-resistance, the textiles being impregnated with the agent and then heat cured. C. O. C.

Improving the Resistance to Creasing of Textiles containing Cellulose Acetate. Celanese Corpn. of America. *BP* 730,111

Impregnation with $> 0{\cdot}625\%$ of an elastomer, e.g. by impregnation with a latex, not only imparts resistance to creasing but also imparts a fuller handle without any tendency to stiffness. C. O. C.

Gas Fume Fading Inhibitor. Celanese Corpn. of America. *BP* 731, 783

Polymers of a dialkylamino alkyl ester of acrylic acid or of an α-subst. acrylic acid are excellent gas fume fading inhibitors, e.g. cellulose acetate yarn dyed with 0·2% 1:4-di(methylamine)anthraquinone is impregnated with 1% of polydimethylaminoethylmethacrylate from aqueous solution on the pad. C. O. C.

Rendering Cellulosic Materials Fire Resistant with Phosphorus Oxychloride–Anhydrous Ammonia Reaction Products. Monsanto. *USP* 2,661,311

The product obtained by treating phosphorus oxychloride with anhydrous ammonia at $< 100°$c., preferably in an inert solvent, is heated to 110–150°c. and freeing the resulting product from NH_4Cl. This is best done by extracting the reaction product by means of solvents, preferably liquid anhydrous ammonia, which dissolve out the NH_4Cl. Cellulosic materials impregnated with the product are given a permanent flameproof finish without loss of handle or strength. C. O. C.

Rendering Textiles Flame-resisting with Polyphosphonitrile Ester. U.S. Secretary of Agriculture. *USP* 2,681,295

The products obtained by treating mixed polyphosphonitrilic chlorides with a chloro- or bromopolyhalogenopropyl alcohol in presence of pyridine are applied to textiles from solution or aqueous dispersion, after which the impregnated textiles are dried and baked. Thus mixed polyphosphonitrilic chlorides (1 part) dissolved in pyridine (5) were mixed with 2:3-dibromopropyl alcohol and kept at 27°c. for 24 hr. A piece of 80 sq. cotton cloth was impregnated with its own weight of the reaction mixture and heated for 30 min. at 110°c. A vertical strip of the treated fabric would not support combustion when lit at the bottom. C. O. C.

Fire-resisting Finish. Titan Co. *BP* 731,176

The material is impregnated with an aqueous solution of tetravalent titanium chloride and a phosphate soluble in such a solution, partly dried so as to form a transparent gel on the material and finally treated with an alkali, washed and dried. The finish is fast to washing and does not affect the appearance, texture or tensile strength of the material. C. O. C.

Flame-resisting Mixed Halogeno Phosphate–Lower Aliphatic Ester of Cellulose. Celanese Corpn. of America. *USP* 2,678,309

Organic derivatives of cellulose containing free hydroxyl groups can be rendered completely and permanently non-flammable by treatment with a halogenoalkyl ester of halogeno phosphoric acid, e.g. bis-(2'3-dichloro-propyl)-chlorophosphonate. C. O. C.

Tenter Frame Washing System for Mercerising Ranges. Springs Cotton Mills. *USP* 2,678,483

The wash water is recirculated independently at a number of points in parallel from a common supply having a relatively low throughput. This gives much more effective washing than previous methods and the wash water finally discharged contains 10% of alkali. C. O. C.

Reducing the Felting Power of Wool. British Industrial Plastics. *BP* 730,872

An aminotriazine-aldehyde condensate or precondensate, e.g. a melamine-formaldehyde condensate, is incorporated in the material which is then treated with aqueous acid at or near the boil. C. O. C.

Reducing the Felting Power of Wool with Substituted Polymers of Ethylene. DuP. *USP* 2,678,287

The materials are treated with a high mol. wt. substituted hydrocarbon polymer or like addition agent and if desired a fixative. Thus wool is impregnated with 2–10% by wt. of a chlorosulphonated polyethylene and an *ω*-diamine or -diamide of 2–6 C and baked at 150°c. for 3 min. C. O. C.

Shrinking Woollen and Worsted Fabrics. James Bailey (Engineers). *BP* 732,055

The fabric is dampened with steam and/or water and is then carried in an inclined conveyor through a drying chamber to a drying cylinder. The conveyor is formed of transverse supports between which the fabric falls in festoons so that it is carried through the drying chamber in absence of tension. C. O. C.

Imparting Patterns to the Nap on Cloth. M. Decloux. *BP* 731,305

Cloth having a nap is treated with scraping blades which penetrate to the base of the nap and gather it up from its bottom into crests and so form reliefs or undulations according to the position of the blades. C. O. C.

Reducing the Plastic Extensibility of Synthetic Linear Polymer Textiles. H. Weikert. *BP* 729,580

The cold-drawn filaments or yarns or fabrics made from them are stretched for > 5 min. at 30–60°C. below the melting point of the polymer. W. G. C.

Thin Coated Fabric of High Resistance to Tearing. United States Rubber Co. *BP* 732,048

Ribbon-like yarns of < 70 denier each containing little twist and composed of a number of continuous filaments are woven loosely into fabric. Because of their low twist the yarns lie very flat in the resulting fabric which is very thin but well covered and strong. It is then coated, e.g. rubber proofed, to yield a very light weight waterproof fabric that is highly resistant to tearing. C. O. C.

Coating Textiles and Other Materials with Resins obtained from Sulphur Dioxide and Unsaturated Organic Compounds. Phillips Petroleum Co. *BP* 731,359

The unsaturated compound, e.g. 1-butene, is treated in aqueous emulsion with SO_2 in presence of catalysts and emulsifying agents. The product after being stripped by steam of all volatile impurities is used for coating, impregnating or laminating textiles, paper, etc., e.g. for the rubber-proofing of textiles. C. O. C.

Non-woven Fabric. Steralon Process International. *BP* 730,251

A fleece of randomly distributed fibres is impregnated with sodium carboxymethyl cellulose and then heated to 300–450°C. to insolubilise the cellulose derivative.

BP 731,753

Modification of *BP* 653,680 (J.S.D.C., **67**, 356 (1951)) the carded lap being impregnated with both solutions.

Non-woven Fabric. A. J. Auchterlonie. *USP* 2,681,866

A carded lap or the like is coated on one or both sides with a thermoplastic resin, heat pressed to fuse the resin to the surface fibres and cooled. C. O. C.

Waterproof Laminated Sheet Material. B. Messing. *BP* 730,052

An imperforate flexible waterproof laminated sheet comprises two outer layers of self-sustaining moisture-resistant thermoplastic material which can be repeatedly softened by heat to a consistency enabling it to permeate porous wadding and hardening on cooling, and an intermediate layer of porous padding. The two outer layers are fused together by heat and pressure through the wadding at selected points. C. O. C.

Water-soluble Polyvinyl Alcohol Fibres. Cluett, Peabody & Co. *BP* 731,153

Treating water-soluble polyvinyl alcohol fibres with boric acid in anhydrous solution but without rendering them brittle makes them resistant to the plasticising and tackifying influence of atmospheric moisture and to elongation under tension but does not affect their solubility in water. C. O. C.

Laminated Fabrics. British Nylon Spinners. *BP* 730,042

When a polyamide fabric is moistened with a phenol dissolved in solvents having little swelling action on polyamides, e.g. phenol in *cyclo*hexane, very good adhesion is obtained if it is hot pressed between two polyamide or polyamide/cotton fabrics. C. O. C.

Stiffened Washable Garments. Carl Freudenberg. *BP* 729,569

A stiffened washable garment which is crease-resistant and permeable to air and water comprises an inner and outer layer of woven washable fabric, and secured to one or both of them an intermediate layer of a fabric-like, porous, permeable, flexible, crease- and shrink-resistant material. This material consists of an open skeleton of randomly intermingled fine fibres and is impregnated with small, filmy lamellar particles of a flexible, solid, thermoplastic binding agent. C. O. C.

Laminated Fabrics. FBy. *BP* 731,181

A layer of an *iso*cyanate-modified polyester is bonded to one side of a fibrous interlayer the other side of which is bonded to a layer of another elastomer or synthetic thermoplast. C. O. C.

Non-woven Pile Fabric. N.V. Koninklijke Vereenigde Tapijtfabrieken. *BP* 730,269

When non-woven pile fabric is produced by feeding thread or fibrous webs between lamellae fixed to a rotating drum, followed by applying a coating of hardening material to the projecting folds of the inserted threads, there is a danger that some of the folds will be pulled out before the coating material has set or even been applied. This is overcome by incorporating into the thread-inserting device a further device whereby thin strips are inserted immediately after the threads in such a way that they lie inside the folds, sufficiently firmly to prevent the folds being pulled away. The strips can be withdrawn sideways after the coating material has been applied and hardened. J. W. B.

Action of Chlorine-liberating Detergents on Crease-resist Finishes (VII p. 480).

Piece Dyeing and Finishing of Polyacrylonitrile Goods—II (VIII p. 480).

Improving the Fastness of Colouring Matters in Textiles and Paper (VIII p. 481).

Dyeing and Finishing Hosiery (VIII p. 481).

XI—PAPER AND OTHER CELLULOSIC PRODUCTS

Preparation of Cellulose Trinitrate. C. F. Bennett and T. E. Timell. *Svensk Papperstidning*, **58**, 281–286 (30 April 1955).

In a study of the possibilities of preparing a completely substituted cellulose nitrate with the same D.P. as the original cellulose, it was found that N_2O_5 did not give the maximum N substitution and, when used alone, caused severe degradation. The presence of BF_3 in the HNO_3–H_3PO_4–P_2O_5 mixture did not increase its nitrating power. A mixture of HNO_3 + CH_3COOH + $(CH_3CO)_2O$ (43 : 32 : 25) nitrated purified cotton linters and ramie celluloses at 0°C. to the maximum nitration level (14·14% N) with no apparent degradation within the first few hours. The reaction was slow, and prolonged nitration caused a considerable decrease in the D.P., probably owing to decomposition of the unstable nitration mixture. S. V. S.

Manufacture of Sodium Carboxymethylcellulose. Kh. U. Mukhammedov. *Doklady Akad. Nauk Uzbek. S.S.R.*, (12), 29–30 (1953): *Referat. zhur.* (*Khim.*), No. 29889 (1954): *Chem. Abs.*, **49**, 4285 (25 March 1955).

An improved method for the preparation of Na carboxymethylcellulose from alkali wood cellulose and chloroacetic acid is described. A. E. S.

Chemical Synthesis of Glucose Polymers. W. J. Whelan and S. Haq. *Chem. and Ind.*, 600 (21 May 1955).

Condensation of halogeno-sugars with hydroxylic compounds in the presence of silver oxide or carbonate yields a β-glycoside (Koenigs–Knorr reaction). If the halogen and the hydroxyl group are present in the same monosaccharide, controlled polymerisation of a sugar unit is possible. 2:3:4-Tri-*O*-acetyl-1-bromo-α-D-glucopyranose, treated in alcohol-free chloroform at 2°C. with silver oxide, iodine, and anhydrous $CaSO_4$, polymerised in the manner suggested. Intramolecular elimination of HBr also occurred with the formation of tri-*O*-acetyl-laevoglucosan. Products of the reaction were removed at intervals, deacetylated, and examined by paper chromatography. Gentio-biose, -triose, and -tetraose were formed within one day. After 3 days pentose and hexose were detected. Possible applications to the synthesis of polysaccharides are suggested. W. R. M.

PATENTS

Paper of Improved Wet Strength. Allied Chemical & Dye Corpn. *USP* 2,680,682

The resins obtained by reacting HCHO with urea or melamine and an aliphatic carboxylic acid of < 9 C, not > 4 C per carboxy group and substituted by NH_2, OH or COOH are added to the pulp to impart high wet strength to the resulting paper. C. O. C.

Sizing Paper. Allied Chemical & Dye Corpn. *USP* 2,680,073

Silanols of formula—

$$R_mSiO_n(OH)_{4-(m+2n)}$$

(m = 0·05–3·0; n = 0–1; R = same or different organic radicals of 1–12 C), e.g. ethyltrichlorosilane, are added at the wet-end of the paper-making process, the web being brought to pH 3–8 within 5 min. of the silanol being added. C. O. C.

Glossy Mineral-coated Paper. S. D. Warren Co. *USP* 2,678,890

The freshly coated side of the paper is pressed against a polished casting drum by means of a backing roll, the entering side of the nip between the drum and the paper being kept continuously flooded with water. This eliminates the possibility of air being entrapped in the nip and so ensures a flawless coating. C. O. C.

Manifold Material. National Cash Register Co. *BP* 731,443

Incorporating individual cellulose fibres into the transfer coating of the pressure-rupturable kind, e.g. that described in *BP* 646,926 (*J.S.D.C.*, **67**, 128 (1951)) prevents smudging of sheets placed next to the coating. C. O. C.

Benzene Hexachloride as Extender for Cellulose Acetate. Olin Mathieson Chemical Corpn. *BP* 731,462

Up to 25% of benzene hexachloride may be incorporated into cellulose acetate without deleteriously affecting the properties of the cellulose acetate. C. O. C.

Transparent Flame-resistant Cellulose Acetate Butyrate. Diamond Alkali Co. *USP* 2,680,691

Incorporation into cellulose acetate butyrate of a highly chlorinated aliphatic hydrocarbon wax made from a hydrocarbon of mol. wt. within the range of C_{18}–C_{26} hydrocarbons and containing 64–68% chemically combined chlorine makes it flame-resistant while preserving its transparency. C. O. C.

Dyeing Lignocellulose Hardboards (VIII p. 481).
Factors influencing the Results obtained by Fractional Precipitation of Cellulose Nitrates (XIV p. 488).

XII—LEATHER; FURS; OTHER PROTEIN MATERIALS

Staining of Aniline-dyed Leathers. *Leather Trades Rev.*, 114, 568 (1954): *J. Amer. Leather Chem. Assocn.*, 50, 156 (March 1955).

Shoe uppers, made with aniline-dyed leather, are prone to stain if shoe linings are made of cloth containing more than 5% organic solubles (by hot extraction), 3% organic solubles (by warm extraction), and 1% soluble inorganic salts. To keep aniline-dyed leathers clean, it is recommended that a thin, continuous wax film be spread over the surface of the leather. C. J. W. H.

Degradation of Collagen. II—The Solubilisation Process in the Acid pH Range. A. Veis and J. Cohen. *J. Amer. Chem. Soc.*, 77, 2364–2368 (5 May 1955).

Acid dissolution data suggest that collagen fibres break apart into more or less discrete units depending upon the pH of extraction. Less than 1·0 m.mole of new free terminal amino groups per 100 g. of residual protein are formed during dissolution. Thus the average mol.wt. of the soluble fragments must have been > 100,000 at the start if dissolution involves hydrolysis of peptide bonds. The soluble portion obtained under the least drastic conditions contains less N than the insoluble portion, indicating that their amino acid content differs. The N content of both portions changes regularly according to the amount of solubilisation and this supports the view that intact collagen is composed of a series of related proteins of similar but not identical composition. Some of the N lost during acid extraction may be due to hydrolysis of the asparagine and glutamine terminal amide residues and exposes carboxyl groups. Along with the slight chemical changes, heating, quantitatively independent of pH, causes reorientation of the functional groups in the fibre. This change manifests itself in the increased affinity for dyes. The effective pK of some of the titratable groups is altered during heating. This reorientation of the fibrous network may be the first step in the solubilisation of collagen in acid liquors. C. O. C.

Degradation of Collagen. III—Characterisation of Soluble Products of Mild Acid Degradation. A. Veis, D. N. Eggenberger, and J. Cohen. *J. Amer. Chem. Soc.*, 77, 2368–2374 (5 May 1955).

The soluble protein obtained by mild hot acid dissolution of purified cow-hide collagen which has not been pretreated does not behave as a typical gelatin but is more closely related to native collagen and "procollagen". C. O. C.

Theory of Protein Solutions. T. L. Hill. *J. Chem. Physics*, 23, 623–636 (April 1955).

The McMillan–Mayer solution theory is applied to protein solutions taking into account the binding of solvent or other molecules to the protein. W. R. M.

PATENTS

Washing and Soaking Skins and Hides. Dow Chemical Co. *BP* 730,697

Washing and soaking skins in an aqueous bath containing diethanol glycine, ethylenediaminetetraacetic acid, propylenediaminetetraacetic acid, trimethylenediaminetetraacetic acid, 1:2-diamino*cyclo*hexanetetraacetic acid, mono(hydroxyethyl)ethylenediaminetetraacetic acid or monobenzylethylenediaminetetraacetic acid or an alkali metal or amine salt thereof greatly facilitates subsequent processing and results in an improved leather. C. O. C.

Preventing Bacterial Damage when Softening Skins or Hides. Boehme Fettchemie. *BP* 730,895

One or more antibiotics, preferably having a polypeptide structure, e.g. actinomycin, aureomycin or penicillin, are added to the softening liquor. C. O. C.

Staking Hides and Skins. British United Shoe Machinery Co. *BP* 731,164

Hides and skins are staked by treating them across their full width with bladed rollers. C. O. C.

Rendering Leather Water Repellent. Dow Corning Corpn. *USP* 2,678,893

The leather is impregnated with a methylsiloxane composed of trimethylsiloxane units and SiO_2 units in such proportions that CH_3:Si::1:1–2·5. C. O. C.

Simple Rapid Method for determining the Dyeing Behaviour of Anionic Dyes on Chrome Leather (XIV p. 488).

XIII—RUBBER; RESINS; PLASTICS

Synthesis of Esters of Polyvinyl Alcohol. S. N. Ushakov and T. A. Kononova. *Izvestiya Akad. Nauk S.S.S.R., Otdel. khim. nauk*, 117–125 (Jan.–Feb.). **Some Physicomechanical Properties of Esters of Polyvinyl Alcohol.** Idem. *Ibid.*, 335–343 (March–April 1955).

Esterification of portions of a given sample of polyvinyl alcohol (sp. viscosity 0·74) yields formic, acetic, propionic, butyric, and *iso*butyric esters varying in degree of substitution (DS) from 30 to 100%, but almost constant in average chain length (this is verified by viscosity measurements on the polyvinyl alcohol recovered by hydrolysis). Films of the preparations are subjected to physical tests in the vitreous and highly elastic states, and their vitrification temp. T_g are determined (30, 30, −2, −6, and 15°c., respectively, for the fully substituted esters). For all esters except the formates, T_g is const. over the range DS = 100–70% and rises at DS < 70% to the value of 85°c. for polyvinyl alcohol. For the same esters the tensile strength and the elastic modulus increase constantly as DS falls, but, in the highly elastic state, the extension at break is constant over the range DS = 100–30%. The formic esters have minimum T_g and minimum tensile strength at DS = 40–50%. A. E. S.

PATENTS

Granules coated with Pigment. N.V. Onderzoekingsinstituut Research. *BP* 730,600

Granular material, especially synthetic thermoplastic polymers, is heated with a pigment in presence of 0·05–0·2% (on the wt. of polymer) of a compound of formula

$R^1{\cdot}X{\cdot}R^2$ (R^1 = strongly hydrophobic group containing a hydrocarbon radical of > 9 C; R^2 = strongly hydrophobic group containing at least one group of formula $(CH_2CH_2O)_n$ ($n = > 3$); X = group connecting R^1 and R^2 by main valencies), e.g. Berol Emu O3, $C_{12}H_{25}O(CH_2CH_2O)_{10}CH_2CH_2OH$. The product does not change in colour when kept or shaken about, e.g. in transit. It is suitable for melt-spinning or injection moulding. C. O. C.

Treating Polymethacrylates with Ionising Radiation. United Kingdom Atomic Energy Authority. *BP* 730,476

When polymethacrylates are subjected to penetrating ionising radiation they change colour slightly and small bubbles gradually form within them, the bubbles rapidly becoming larger and more numerous until the whole becomes cellular and finally powders. By irradiating below the softening point of the polymers no bubbles are formed in a surface layer and by suitable selection of the irradiation dose and temperature of subsequent treatment a bubble-free outer skin is formed within which a strong cellular mass develops, the skin setting hard and smooth. C. O. C.

High Energy Irradiation of Polymers. United Kingdom Atomic Energy Commission. *BP* 732,047

Linear polymers, e.g. polythene, can be made more resistant to organic solvents by subjecting them to ionising irradiation in amount insufficient to cause any great change in their mechanical properties when measured at ordinary temperature. Mechanical properties at high temperatures may be affected, a rubber-like resistance at such temperatures being imparted in some cases. Substances which had a marked m.p. before treatment no longer melt and flow, but may still soften when heated. C. O. C.

Processing Glass Fibre Compositions. Hawley Products Co. *BP* 730,054

Cut bundles of glass filaments are mixed in water with a fusible resin capable of being cured to an infusible state and which has an affinity for glass filaments. It is added to the water either just before or just after the filament bundles and serves to prevent them disintegrating; it is cured while the mixture is wet and thus gives the fibrous mass sufficient cohesion for further handling, e.g. preforming, to be possible. Further resin-impregnation and heat-hardening to the final moulded article can then be carried out. The process is suitable for mixtures of glass rovings with any other fibrous pulp material. J. W. B.

Macromolecular Compounds. LXIX— Dependence of the Properties of Polyamides on the Extent of Hydrogen Bonding. LXXIII— Some Two-component Mixed Polyamides (VI p. 479).

Linear Polyamides from Pyrrolidone (VI p. 479).

Acetone-soluble Acrylonitrile-Vinylidene Chloride Copolymers (VI p. 480).

Improving the Dyeing Properties of Polyacrylonitrile (VI p. 480).

XIV—ANALYSIS; TESTING; APPARATUS

Wool Grease. Investigation of a Method of Identification in Presence of Processing Oils. C. Schirle, C. Allabert, and G. Gianola. *Bull. Inst. Text. France*, (52), 41–52 (April 1955).

A modified Liebermann–Burchard reaction was used to determine cholesterol (I) and "*iso*cholesterol" (lanosterol, dihydrolanosterol, γ-lanosterol, and agnosterol) (II) as follows— (*a*) colour due to I was measured at $\lambda = 620$ μ.; (*b*) test solution diluted to give a I content of 50 mg./litre and colour due to I and II measured at $\lambda = 495$ μ., using a calibration curve for II in presence of a constant amount of I. Satisfactory results were obtained on known mixtures of I and II. In analysis of mixtures of I and II and various commercial oils, the calc. amount of II was found to be affected by oil present. Accurate results were obtained for I except in presence of products containing a high proportion of oleine or vegetable oils. The following factors must be taken into account if the sterol content of wool grease is to be used for its estimation— (1) Analysis of grease from 10 wools of different origins gave contents of I ranging from 11 to 19% and of II ranging from 3 to 12%. (2) Investigation of composition of grease at different stages of scouring showed a marked decrease in % of I and a still more marked decrease in % of II as scouring proceeded. (3) A sample of wool grease kept exposed to air showed a marked decrease in II content with time. Storage under N_2 and refrigeration prevented this decrease. This factor may be at least as important as place of origin in determining the relative amounts of I and II in the grease from different wools. It is suggested that an approximate value for the amounts of wool grease and processing oil in extracts of wool may be obtained from a determination of I in the unsaponifiable fraction, assuming a mean value of 10–12% for the I content of the grease in scoured wool. J. C. F.

Adsorption and Chromatography of Fatty Acids on Charcoal. J. Cason and G. A. Gillies. *J. Org. Chem.*, 20, 419–427 (April 1955).

Luminol Indicator Paper for Detection of Hydrogen Peroxide. A. A. Ponomarenko. *J. Anal. Chem. U.S.S.R.*, 10, 132–133 (March–April 1955).

Filter paper is impregnated with a 0·5% soln. of luminol (cyclic hydrazide of 3-aminophthalic acid) in 0·1 N-NaOH, placed for a few min. over formic acid (the vapour of which neutralises the alkali and enables the paper to be kept indefinitely), and dried. A drop of the soln. to be tested, a drop of catalyst (soln. of 0·1 g. of CuCl in 100 ml. of 1% NH_3 soln.), and a drop of dil. alkali are applied successively to the paper. When H_2O_2 is present, there is a bright chemiluminescence (detectable with a 0·0005% soln. of H_2O_2). A. E. S.

Characterisation of Alkylbenzenes. H. Pines and A. W. Shaw. *J. Org. Chem.*, 20, 373–377 (March 1955).

A method employing Friedel–Crafts acetylation and subsequent conversion into the 2:4-dinitrophenylhydrazone or semicarbazone is described, the results for 69 hydrocarbons being tabulated. H. E. N.

Partition Chromatography of Tannins and Pigments XXIII— Paper Chromatography of Tannins. 1. Fundamental Conditions. Y. Oshima, T. Nakabayashi, N. Hada, and S. Matsuyama. *J. Agr. Chem. Soc. Japan*, 28, 618–621 (1954); **XXIV— Paper Chromatography of Tannins. 2. Classification of Tannins by Paper Chromatography.** Y. Oshima, T. Nakabayashi, and N. Hada. *Ibid.*, 621–629: *Chem. Abs.*, 49, 7065 (25 May 1955).

XXIII— 0·5% $FeCl_3$ in methanol was the best colour reagent and observations under ultraviolet radiation is useful. Filter paper should be washed with dil. HCl or treated with 1–2% H_3PO_4 or oxalic acid for 30–60 sec. Of many developing solvents tested the following were satisfactory— the upper and lower layers of butyl alcohol : acetic acid : 1% aq. $NaHSO_3$ (4 : 1 : 5 by vol.), phenol saturated with 1% aq. $NaHSO_3$, and 1% aq. $NaHSO_3$ saturated with phenol. These solvents could be used for two-dimensional chromatography. Pretreatment of the paper with 2% H_3BO_3 for 1 min. increased the separation of individual components.

XXIV— Using two-dimensional paper chromatography five types of patterns occurred in the chromatogram— (1) typical pattern of the depside tannins, e.g. Chinese gallotannin and *Spirogyra* tannin, (2) a type of myrobalan tannin, (3) a type of phlobatannin, the tannins of wattle bark, *Myrica rubica* bark, and gambir extract, (4) the tannin of mangrove extract and chestnut bark, and (5) the tannin of tea leaves and apples, which consist of lower polyphenol derivatives. C. O. C.

Analytical Application of Reflectancy Determination. I— Filter Paper Determination of Spectro-reflectancy of Coal Tar Colours for Foodstuffs. K. Yamaguchi, S. Fujii, T. Tabata, and S. Kato. *J. Pharm. Soc. Japan*, 74, 1322–1326 (1954).

The visible reflectance spectra of 24 food dyes spotted on filter paper were measured with a Beckmann Spectrophotometer Model B having a diffuse reflectance attachment. Most of the spectra so obtained agreed well with the absorption of the solutions of these dyes. A mixture of dyes was separated by paper chromatography and the reflectance of the separated coloured spots measured. The values obtained agreed well with those of the single dyes.

II—Determination of Surface Reflectancy of 2:4-Dinitrophenylhydrazones. K. Yamaguchi, S. Kukushima, T. Tabata, and M. Ito. *Ibid.*, 1327–1332.

2:4-Dinitrophenylhydrazones of 3 aliphatic and 8 terpenoid carbonyl compounds, furfural, 5-nitrofurfural, 11 aromatic and 8 aromatic ketones were studied. Their surface colours were compared by photo-reflectance at 4000–7000 A. using the diffuse attachment. Reflectance ratio in the different wavelengths and saturated reflectance were compared. Comparison of the characteristics of the slope and the wavelength at the standing point of the reflectance curves made possible representation of slight differences in colour.

III—Notation of Surface Colour of 2:4-Dinitrophenylhydrazones on C.I.E. Colour Chart. K. Yamaguchi, M. Ito, and K. Bando. *Ibid.*, 1332–1335.

The surface colour (data of spectroreflectance) of 30 2:4-dinitrophenylhydrazones was plotted on the C.I.E. colour chart. This enabled 3 colour coefficients, brightness, dominant wavelength and purity of colour to be determined.

IV—Colours and Absorption Maxima of 2:4-Dinitrophenylhydrazones in Alkaline Solution. K. Yamaguchi, S. Fukushima, T. Tabata, and M. Ito. *Ibid.*, 1335–1338: *Chem. Abs.*, **49**, 4441 (10 April 1955).

Absorption maxima of 32 2:4-dinitrophenylhydrazones in acetone are given and compared with those obtained if an alkali, e.g. 1% alcoholic caustic soda, pyridine or ammonia, is added. Some observations were made about the relationship between the characteristic colour tone and chemical structure. C. O. C.

Identification by Paper Chromatography of Dyes in Cosmetics. J. Deshusses and P. Desbaumes. *Mitt. Lebensm. Hyg.*, **45**, 484–489 (1954): *Chem. Abs.*, **49**, 5781 (25 April 1955).

Methods for identifying organic dyes in hair tints, hair dyes and dye shampoos, are described C. O. C.

Photoelectric Estimation of Fluorescent Brightening Agents on Cloth. J. Pinte and P. Rochas. *Melliand Textilber.*, **36**, 362 367 (April 1955).

The apparatus used should permit three different types of measurement—(*a*) intensity and hue of the fluorescent effect; and the signatures (*b*) including and (*c*) excluding the fluorescence. A modified T.C.B. photoelectric colorimeter is described S. R. C.

Rapid Volumetric Determination of Antimony, Copper, or Iron in Textile Materials. A. G. Hamlin. *J. Textile Inst.*, **46**, T247–T255 (April 1955).

Determination of Sb has become increasingly important through the use of Sb-containing flameproof finishes, and an attempt to find a suitable method has led to the discovery that Fe and Cu can be determined in the same way. After digestion of the textile material in a mixture of nitric, sulphuric, and perchloric acids, the metals present are reduced to their lower valency states by the action of $TiCl_3$ in hot dilute perchloric acid solution. Excess of reductant is oxidised by the perchloric acid, which does not affect the reduced metals, and these are then determined by titration with suitable oxidising agents. Sb and Cu, and Sb and Fe, can be determined in presence of each other, but Cu and Fe cannot separately be determined if both are present J. W. B.

Determination of Vegetable Matter in Woolly Burr removed at the Card. R. P. Harker, J. L. Horner, and F. O. Howitt. *J. Textile Inst.*, **46**, T256–T263 (April 1955).

Examination of published methods for the determination of vegetable matter in burry wools containing a high percentage of burr shows that only dissolution in NaOH affords a reliable technique. Serious errors can arise because of inadequate sampling, but samples of 1–2 g. can be satisfactorily taken by grinding wool-burr mixtures with an electrically driven wheel faced with emery paper; alternatively, excellent results can be obtained by using a rotary cutter. Sand and seeds are troublesome and should be separated first. The papain–bisulphite–urea method is examined and considered unsuitable. A rigidly controlled method entailing 2 min. boiling with 10% NaOH is described and gives a very consistent recovery of burr, but the fact that a correction factor of 150% must be applied suggests that the procedure is not ideal. The correction factor is finally eliminated in an entirely different method, in which the samples are dyed to saturation with Solway Blue BNS, the concentration of which is measured photoelectrically. The relation between % dye adsorbed per gram of mixture and the proportion of wool is linear, and good agreement is found with the NaOH method. It is considered that the procedure may be adaptable to the quantitative determination of binary fibre mixtures; preliminary experiments give satisfactory results with wool–nylon and wool–Terylene. J. W. B.

Cause of Variability of Ether Extracts of Soap-scoured Wool and Top. D. P. Veldsman and R. C. Palmer. *J. Textile Inst.*, **46**, P 286–P 289 (April 1955).

Experimental evidence is presented which suggests that interlaboratory differences in the ether extract of soap-scoured wool and top may be due to variable regain. Small amounts of moisture in the wool are slowly removed by ether in the course of Soxhlet extraction, but before they are removed are capable of hydrolysing some of the soap present and thus leading to an artificially high oily extract. It is suggested that wool should be thoroughly dried prior to extraction, and that precautions should be taken to avoid drawing-in of air during the extraction cycles. J. W. B.

Apparatus for Treatment of Cotton Fabrics on a Laboratory Scale. W. T. Schreiber and A. C. F. Mason. *Amer. Dyestuff Rep.*, **44**, 288 (25 April 1955).

Comparative Solubilities of Natural and Man-made Fibres. S. S. Praeger. *Amer. Dyestuff Rep.*, **44**, 281–282 (25 April 1955).

It is shown that Orlon and Acrilan are both completely soluble in 66·7% $ZnCl_2$ solution, but that only Acrilan is soluble in a solution of 57·1% strength. Further solubility studies are made on a range of 16 fibres and 11 solutions or solvents other than H_2O, and a chart is presented from which the correct procedure for fibre analysis may rapidly be selected. J. W. B.

Determination of Sulphide Sulphur and Reducible Sulphur in Viscose Rayon Materials. T. Kleinert, F. Sieber, and A. Apuchtin. *Textil-Rund.*, **10**, 12–16 (Jan. 1955).

An improved, simple procedure for the differentiation of the various forms of sulphur in viscose rayon has been devised. Sulphide S is determined (in a special apparatus) by conversion to H_2S with HCl, reducible S by conversion to H_2S with HCl–$SnCl_2$. The H_2S is determined colorimetrically as the molybdenum blue complex. Total S is determined by oxidation with alkaline H_2O_2, ashing, and pptn. as $BaSO_4$. Investigation of various samples of viscose rayon showed that on storage sulphide S and reducible S undergo oxidation to H_2SO_4, which, particularly under the influence of light, causes secondary fibre damage. Peroxidic impurities and degradation products of the fibre play an important part in the oxidation reaction, which is catalysed by TiO_2 present in delustred fibres. J. C. F.

Standardised Dissolution of Standard Viscose in the Laboratory. E. Treiber, O. F. Fex, J. Rehnström, and M. Piova. *Svensk Papperstidning*, **58**, 287–295 (30 April 1955).

A dissolving apparatus in which 4 different experimental viscoses can be prepared simultaneously is described, and the influence of different factors on the filter-value is discussed. Particular attention should be paid to the temperature of the dissolving lye and in the dissolving vessels, and the mechanical details, e.g. position of the agitator, have some effect. The speed and time of agitation are important only when very low. Mechanical dissolution takes care of microscopical inhomogeneities, whilst the afterdissolution during ripening affects the submicroscopic inhomogeneities in the viscose. S. V. S.

Micro-reactions for Regenerated Protein and Alginate Fibres. W. Bobeth. *Faserforsch. und Textiltech.*, **6**, 20–25 (Jan. 1955).

Visual effects of the treatment of regenerated protein (groundnut, casein, zein) and alginate fibres with H_2SO_4 at various concn. are illustrated by photomicrographs. Some of the effects are useful for identification purposes. A. E. S.

Regularity of Fabrics measured by means of Photoelectric Colorimetry. A. Barella, C. Pujol, and J. Cegarra. *Bull. Inst. Text. France*, (52), 59–63 (April 1955).

A photoelectric colorimeter was used to measure (*a*) transparency, (*b*) reflectance against a white background, and (*c*) reflectance against a black background at a large number of randomly selected areas on each of several fabrics. A greater degree of dispersion was found in (*b*) than in (*c*), due to reflection from the background: (*b*) was therefore rejected. Factors (*a*) and (*c*) were measured on samples of the same fabric dyed to different colours. For (*a*), the dispersion increased as the depth of colour increased, while (*c*) showed the opposite trend. No significant correlation was found between (*a*) and (*c*).
J. C. F.

Identifying Cellulose Triacetate in Blended Fabrics and Fibres. Anon. *Canadian Textile J.*, **46**, 58–59 (25 March 1955).

Arnel and acetate rayon are sol. in a mixture of methylene chloride and methanol or ethanol (90 : 10), whereas other fibres are not affected; dynel is softened and gains in weight, and should be absent if accurate results are required. Arnel, whether heat-treated or not, may be separated from acetate rayon by immersion in benzyl alcohol for 1 hr. at 50°c., the acetate being dissolved. Full details of the procedure are given.
J. W. D.

Adsorption of Brazilwood and Logwood Colouring Matters by Fibres. O. Newsome. J.S.D.C., **71**, 184 (April 1955).

The method of testing logwood products by trial dyeing on patterns of wool mordanted with "oxidising" and "reduced" chrome mordants (see J.S.D.C., **70**, 392 (1954)) is practically obsolete, and only a fully reduced mordant is usually employed. Haematein is determined by direct dyeing on this mordant, and haematein plus haematoxylin by dyeing with addition of predetermined quantities of sodium nitrite and acetic acid.
C. J. W. H.

Third Report of the Fastness Tests Co-ordinating Committee [Standard Methods for Testing Colour Fastness]. Society of Dyers and Colourists, Fastness Tests Co-ordinating Committee. J.S.D.C., **71**, 283–342 (June), 464–465 (Aug. 1955).

Standard methods are given for testing the fastness of coloured textiles to a wide variety of fading agencies. The tests are classified in four groups— proposed I.S.O. (International Standards Organisation) recommendations, I.S.O. tentative tests, tests under consideration by I.S.O., and residual *Second-Report* tests.
C. J. W. H.

Report of Meetings of Subcommittees 1 and 2, Technical Committee 38, ISO. M. L. Staples. *Canadian Textile J.*, **72**, 60–65 (11 March 1955).

The two subcommittees deal with colour fastness, and shrinkage of fabrics in laundering, respectively.
C. J. W. H.

Review of Several Methods of Hydrosulphite Analysis with special regard to Control of Vat Dyebaths. A. Airoldi and F. Marchi. *Amer. Dyestuff Rep.*, **44**, 283–288 (25 April 1955).

Translation of paper published in Italian in *Tinctoria*, **51**, 43–49 (Feb. 1954)— see J.S.D.C., **70**, 528 (1954).

Measurement of the Creasing Angle and Factors influencing the Results. K. Quehl and W. Siegel. *Melliand Textilber.*, **36**, 491–494 (May 1955).

The original Quehl method and a development termed the Broadside method are compared. In the latter a strip 5 cm. × 2 cm. is folded into legs of 1 cm. × 5 cm., covered with a microscope slide, and loaded. After release, the angle is measured with the longer leg laid flat (illustrated).
S. R. C.

Modification of the AATCC Flammability Tester. E. Freedman and D. Chaucer *Amer. Dyestuff Rep.*, **44**, 229–230 (28 March 1955)

An electric automatic timing device is described for the AATCC flammability tester
J. W. B.

Factors influencing the Results obtained by Fractional Precipitation of Cellulose Nitrates. T. E. Timell. *Svensk Papperstidning*, **58**, 234–239 (15 April 1955).

A study of the effect of degree of substitution and refractionation on the results obtained by fractional precipitation of a cellulose nitrate led to the conclusion that complete elimination of the influence of the chemical composition of the nitrate on its fractionation could be achieved only by using a completely trisubstituted product. The possibility of subjecting the solution of cellulose nitrate to a prolonged series of refractionations was also discussed with regard to the significant instability of the solution.
S. V. S.

Ion-exchange Separations on Chemically Modified Cellulose. N. F. Kember and R. A. Wells. *Nature*, **175**, 512–513 (19 March 1955).

Excellent separation of Fe, Cu, and Ni has been obtained on strips of phosphorylated paper, prepared from cotton fabric treated with urea phosphate to give a P content of 3·85% and a capacity of 2·04 m.equiv./g., using 2N NaCl as the developing solvent. The same developing solvent resolved Co and Pb from a mixture containing also Fe^{3+}, Al, and Be, but these latter remained together in the same band. A partial separation of Au, Pt, Pd, and Rh, as their chloro acids, has been effected on papers prepared from cotton fabrics treated with 2-aminoethylsulphuric acid; difficulty was experienced owing to the uneven wetting properties of the paper. A column was prepared from phosphorylated paper pulp blended with untreated cellulose pulp, converted to the acid form with dil. HCl, and then to the Na form by washing with a mixture of NaCl (2N.) and Na_2CO_3 (0·1N.). A mixture of Fe^{3+}, Cu, and Ni was retained at the top of this column; elution with 0·5N HCl removed the Cu and Ni together, but elution with M·$CaCl_2$ removed the Ni and the Cu separately, and in that order, the Fe^{3+} remaining (in both cases) firmly held. A complexing agent such as acetone–HCl was necessary for the removal of the Fe, and this left the column in the acid form ready for re-use.
J. W. D.

Simple Rapid Method for determining the Dyeing Behaviour of Anionic Dyes on Chrome Leather. G. Otto. *Leder*, **5**, 244–246 (1954) : *Chem. Abs.*, **49**, 3538 (10 March 1955).

Place in a test tube 1 c.c. (0·65 g. ± 10%) of standard, weakly chromed hide powder which has been loosened by shaking. Pipet water (10 c.c.) brought up to pH 3·7 with H_2SO_4 into the powder, shake, and add 6 drops (approx. 0·25 c.c.) of an aq. soln. of the dye (5 g./l.). Shake for 30 sec., and filter through glass wool. Refilter (up to 20 times through may be needed) until the filtrate is optically clear and place it in tube *A*. Wash the powder on the glass wool with dil. NH_4OH (1 c.c. of 1% NH_4OH/l.; pH 8·3), repour the filtrate through twice and then place it in tube *B*. Compare the colour of tube *A* with that of a tube containing water at pH 3·7 (10 c.c.) to which 1 drop of the dye soln. has been added. Compare tube *B* with a tube containing NH_4OH (pH 8·3) and 1 drop of soln. Dyes are divided into 8 groups depending on the colours they give in tubes *A* and *B* respectively: 1— none, none; 2— none, up to 30% of control; 3— > 50%, > 10%; 4— > 30%, > 30%; 5— > 30%, > 50%; 6— > 50%, 100%; 7— 50–100%, > 100%; 8— > 100%, > 100%. Penetration of the dyes into chrome leather is at a minimum with group 1 dyes and goes up to a maximum with group 8 dyes. Freshly chromed hide powder does not give as good results as standard hide powder. The method is not suitable for vegetable-tanned leather.
C. O. C.

Evaluation of Organic Dyes for the Leather Industry. V. Pektor and M. Radil. *Československ. kožařství*, **2**, 51–52 (1952): *Chem. Abs.*, **49**, 7280 (25 May 1955).

The Czechoslovakian Leather & Allied Trades Research Institute recommends the following tests— solubility at 20 and 50°c.; dyeing test (on full grain and on suède leather and also after snuffing); fastness to light, acid, and alkali; results obtained with 14 Czechoslovakian dyes are tabulated.
C. O. C.

Existence of Individual Fibrils in Native Fibre Cellulose (VI p. 478).

Formation of Carbonyl Sulphide in Viscose Manufacture (VI p. 479).

Desizing Procedures in relation to Enzyme Evaluation (VII p. 480).

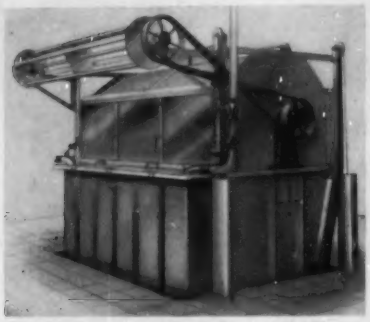
This reproduction of an actual print on delicate nylon illustrates one interesting use.
Though Cotton and Spun rayon provide by far the greatest outlet, ARIDYE pigment colours are successfully applied to almost every type of fibre, both natural and synthetic.

Enquiries to:

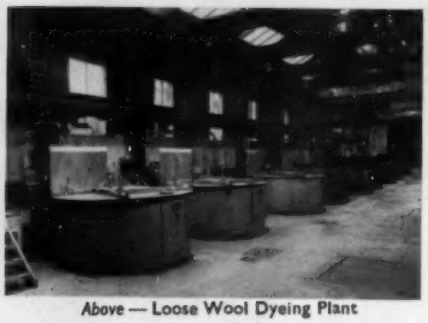
TENNANTS TEXTILE COLOURS LIMITED

35/43, Ravenhill Road, Belfast or to the Area Distributors

Aridye Colours and the use thereof are protected by British Patents 522941, 523090, 524803, 552919, 561641, 561649, 531882.



Above — Enclosed Piece Dyeing Machines



Above — Loose Wool Dyeing Plant

Above — Top Dyeing Machines

Hank Dyeing Machines for Carpet and Hand Knitting Yarns

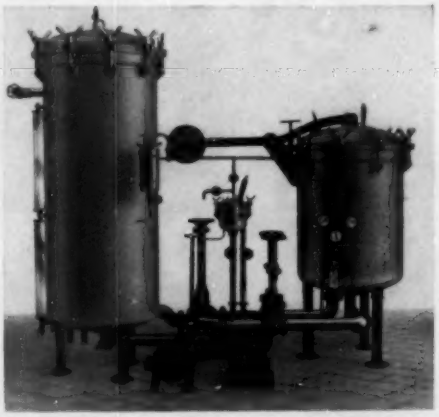
LONGCLOSE DYEING PLANT for the WOOLLEN and WORSTED TRADES

Whatever fibres, natural or synthetic are used in the production of woollen and worsted yarns and fabrics, there is a Longclose machine to dye them.

There is a suitable Longclose Plant to dye these fibres at every stage from raw stock to finished goods.

Longclose all Stainless Steel Plant is used throughout the world for dyeing loose material, slubbing, yarns, piece goods and knitted fabrics.

Write for details of the types illustrated, or if you have special requirements, let us place our wide experience at your service.



Pressure Dyeing Plant for all temperatures up to 130°C. for yarns in cheese and cone, and for dyeing synthetics

LONGCLOSE ENGINEERING Co LTD

BOWMAN LANE WORKS
LEEDS 10 · ENGLAND

Telephone 21978-9

NEW MEMBERS AND JUNIOR MEMBERS

Persons desirous of joining the Society as Ordinary Members or Junior Members may obtain Application Forms from the Secretary, or from the Honorary Secretary of any Section of the Society

NEW MEMBERS

Balicki, Z., 1002, 4th Street, Grand'mere, P. Quebec, Canada

Boyd, J., Tennants Textile Colours Ltd., 35–43 Ravenhill Road, Belfast, N.I.

Crawford, R., 12 Golf Road, Burnside, Rutherglen, Glasgow

Egger, Dr. T. M., Ciba Limited, Basle, Switzerland

Fenn, T., Littlewoods (Mail Order Stores Ltd.), Kershaw Avenue, Endbutt Lane, Crosby, Liverpool 23

Gledhill, G. H., Wm. Brook & Sons (Dyers) Ltd., Slaithwaite, near Huddersfield

Hawksworth, J., 7 Lottie Street, Pendlebury, Manchester

Hung, Y. M., Geigy Trading Co. Ltd., Basle, c/o Jardine Matheson & Co. Ltd., P.O. Box 70, Hongkong, China

Hunt, R. E., Tootal Broadhurst Lee Co. Ltd., 56 Oxford Street, Manchester

Lambert, D. S., 7 Grove Road, Newtown, New Mills, near Stockport, Derbyshire

Lead, W. L., Nottingham and District Technical College, Shakespeare Street, Nottingham

Lee, K. R., I.C.I. Ltd., 7 Donegall Square East, Belfast, N.I.

Paul, J., 140 Smithies Street, Fall River, Massachusetts, U.S.A.

Reynolds, D., 2 Kingsdale Avenue, Bolton, Bradford 2

Sharp, J. K., 4 Pasture Lane, Clayton, Bradford, Yorks.

MEMBERS' CHANGES OF ADDRESS

Ashboult, R. F., formerly of 416 Bury Old Road, Prestwich, Manchester, to 394 Wilmslow Road, Manchester 20

Broadhurst, R., formerly of 89 Caledonian Road, Leeds 2, to Flat 1, 814 Wilmslow Road, Manchester 20

Dixey, J. A., formerly of Brotherton & Co. Ltd., Cattells Grove, Birmingham 7, to Fir Cottage, Bewdley Hill, Kidderminster, Worcs.

Drzewicki, M., formerly of 105 Westminster Road, Morecambe, to 3 Ruskin Drive, Bare, Morecambe, Lancs.

Huntley, R. A. R., formerly of Dent Alcroft & Co. Ltd., Church Lane, Midsomer Norton, to c/o Messrs. Blake & Fox Ltd., Summerhouse Lane, Yeovil, Somerset

Oakes, T., formerly of 24 Neptune Boulevard, Strathmore, Quebec, to c/o North American Cyanamid Co. Ltd., 5550 Royalmount Avenue, Town of Mount Royal, Quebec, Canada

Russell, W., formerly c/o 17 Broadway, Earlsdon, Coventry, to 448 Beake Avenue, Coventry

Trueman, H. N., formerly of 30 Ormonde Park, Belfast, to Lisdalgon House, Saintfield, Co. Down, N. Ireland

ADDRESSES WANTED

Alcada, J. C., formerly of 9 Mornington Villas, Flat No. 4, Bradford 8

Crook, B., formerly of 196 Wentworth Street, W., Magog, Quebec

Crowther, D., formerly of 551 Leeds Road, Thackley, Bradford

Dickinson, K., formerly of 16 Hollycroft, Hinckley, Leics.

Ferguson, T. D., formerly of P.O. Box 106, Altavista, Virginia, Mass.

Green, A. N., formerly of 19 Salcombe Road, Basford, Nottingham

Kesprek, A. E., formerly of 15 Huntley Street, Hamilton Street, Ontario, Canada

Meier, P. W., formerly of Oberwilerstrasse 38, Basle, Switzerland

Morrison, A., formerly of America Woollen Co., P.O. Box 930, Lawrence, Mass., U.S.A.

Nixon, I. G., formerly of Adriaan Goekooplaan, 105 Flat Gebouw Catsheuvel, The Hague, Holland

Standring, Dr. P. T., formerly of I.C.I. Ltd., 1200 St. Alexander Street, Montreal, Canada

Stiassny, K., formerly of 70 Bialik Street, Ramat Gan, Israel

Swift, F. S., formerly of 3 St. Annes Street, Ennis, Co. Clare, Eire

a Viscosa, C. I. S., formerly of Dvozione Centrale degh Etabiliment (Dott. Calandra), Via Sicilia 162, Roma, Italy

Warchol, W., formerly of 14 Sparford Avenue, off Wollaton Road, Nottingham

Yeadon, E., formerly of 18 Benton Park Crescent, Rawdon, Leeds

Patents — Designs — Trade Marks

W. P. THOMPSON & CO

CHARTERED PATENT AGENTS

50 LINCOLN'S INN FIELDS
LONDON WC 2
Holborn 2174

12 CHURCH STREET
LIVERPOOL 1
Royal 3172

SITUATIONS VACANT AND WANTED *etc*

The Publications Committee is prepared to receive Advertisements relating to SITUATIONS VACANT, SITUATIONS WANTED, or MISCELLANEOUS ITEMS for insertion on this page of the Journal. Advertisements of Situations Wanted are gratis to individual members, but must not exceed twenty-four words in length.

All inquiries relating to Advertisements in the Journal should be addressed to THE GENERAL SECRETARY, THE SOCIETY OF DYERS AND COLOURISTS, DEAN HOUSE, 19 PICCADILLY, BRADFORD 1, YORKSHIRE.

Replies may be addressed Box —, THE SOCIETY OF DYERS AND COLOURISTS, DEAN HOUSE, 19 PICCADILLY, BRADFORD 1, YORKSHIRE, where all communications relating to these Advertisements are treated in strict confidence.

SITUATIONS VACANT

The engagement of persons answering these advertisements must be made through a Local Office of the Ministry of Labour or a Scheduled Employment Agency if the applicant is a man aged 18–64 inclusive or a woman aged 18–59 inclusive unless he or she, or the employment, is excepted from the provisions of the Notification of Vacancies Order 1952.

Bradford Education Committee

TECHNICAL COLLEGE, BRADFORD

BRITISH NYLON SPINNERS RESEARCH FELLOWSHIP

APPLICATIONS are invited for a Research Fellowship provided by British Nylon Spinners Limited and tenable in the College for a period of not less than two years. The value of the Fellowship will be £400 to £600 per annum, according to qualifications, and will be for work on the structure of wool and nylon staple fibre fabrics.

Forms of application may be obtained from the Principal, Technical College, Bradford 7.

CALICO PRINTING, DYEING AND FINISHING. Assistant Manager required for British-owned Printworks in Portugal. Applicants, who should be between 35 and 42, must possess a recognised qualification in chemistry or textile technology and have had practical experience in the industry. Good salary offered, with free house. Selected applicants will be interviewed in Manchester. Write, in confidence, to Box "JL/221" c/o 95 Bishopsgate, E.C.2.

IMPERIAL CHEMICAL INDUSTRIES LIMITED, Dyestuffs Division, has a number of vacancies in the Technical Service Department for scientists (men) minimum qualifications Honours Degree in chemistry or equivalent, with or without previous industrial experience and for technologists (men) with experience in the dyestuffs using trades. A wide variety of posts are available with excellent prospects of advancement. Age below 30. Applications in writing to Staff Department, Hexagon House, Blackley, Manchester, 9.

KEEN young qualified textile technician/chemist required to assist in Laboratory development and technical servicing of well-established resin pigment printing and dyeing and copolymer finishing products. Apply in writing, giving full details of qualifications and experience to Personnel Officer, Scott Bader & Co. Ltd., Wollaston, Wellingborough, Northants.

PETROCHEMICALS LIMITED have a vacancy for a Chemist who has specialised in or has industrial experience of Colour Chemistry in the Plastics or other fields. This position offers interest and scope in existing and new processes.

Contributory pension scheme; Non-contributory sickness and accident benefit scheme. Applications in writing, quoting Reference Col/47 should be addressed to the Personnel Manager, Petrochemicals Ltd., Partington Industrial Estate, Urmston, Manchester.

BRITISH RAYON RESEARCH ASSOCIATION

RESEARCH Worker required in the Finishing Department. The work involves the finishing of all types of fabrics made from rayons or blends of rayon with other fibres. Practical experience in the application and assessment of finishes would be an advantage. Superannuation under F.S.S.U. Applicants should possess a Chemistry degree or have had equivalent training. Applications, quoting reference "Finishing", with details of experience and salary required, should be sent to the Appointments Officer, British Rayon Research Association, Heald Green Laboratories, Wythenshawe, Manchester 22.

PIGMENT RESEARCH AND DEVELOPMENT

VACANCIES occur in the Research Laboratories at our Pigment Factory for —

(a) Organic Chemists to fill senior positions for research and development of Pigment Dyes, Lakes and Toners. Previous experience a definite advantage but full consideration will be given to applicants with experience in an allied field.

(b) Research Assistants with O.N.C., H.N.C., or equivalents. Excellent opportunities for the right men.

Written replies will be treated in confidence. Box V77

WELL EDUCATED young man with experience in colour matching wanted by modern upper leather tannery in the Manchester area for their Finishing Department. Applications with full details of age, experience and wage required to Box V78.

SITUATION WANTED

BRITISH business executive, age 39, with fifteen years technical sales and service to British and European colour consuming industries. Seeking opportunity to utilise to greater advantage unique experience and first-class contacts with European consumers and distributors. Academic qualifications, also fluent German and French. Propositions welcomed requiring, energy, initiative and top level responsibility. Box W548

MISCELLANEOUS

BOUND copies of the *Journal*, 1918-1950 for disposal. Box M806

THE SOCIETY OF DYERS AND COLOURISTS

ANNUAL DINNER

will be held on

27th APRIL 1956 at THE MIDLAND HOTEL MANCHESTER

This will be the Diamond Jubilee Year of the Manchester Section of the Society and the Perkin Centenary Year

The Society of Dyers and Colourists

SYMPOSIUM

in

NORTHERN IRELAND

on

'BLEACHING DYEING & FINISHING TODAY'

The brochures have now been issued to all members giving particulars of the above Symposium which will be held at Portrush, County Antrim, from 13 to 17 September 1955. The Headquarters, Northern Counties Hotel, Portrush IS FULLY BOOKED and intending visitors who have not notified their required reservations are advised to make their own arrangements direct, but if in any difficulty reference should be made to the General Secretary of the Society

★ ★

(Photograph Courtesy Messrs. Wm. Hall)

BETTER BE SAFE THAN SORRY

The presence on the fibre of lime soap from a previous operation may give uneven dyeing—hence the necessity for close inspection. Calgon (Albright & Wilson's Sodium Metaphosphate) added to a vat liquor will overcome the effect of lime soap, give a better dispersion of the dye, and prevent the dye riding on the foam of the dye-bath. Dyeings are level and colours brighter. You will find that Calgon is invaluable in the Cotton and Rayon industries, as well as with Woollens and Silks. Write for a copy of "Calgon in the Textile Industry".

CALGON

for better dyeing

ALBRIGHT & WILSON LTD. · *Water Treatment Department*

49 PARK LANE · LONDON · W.1 · *Tel:* GROsvenor 1311 · Works: Oldbury and Widnes

aw161

Telephone Dudley Hill 253 & 254 (Private Branch Exchange) Telegrams: BISULPHITE BRADFORD

J B WILKINSON (CHEMICALS) LTD

Manufacturers of

SODIUM HYDROSULPHITE POWDER and CHEMICALS

FOR DYERS BLEACHERS PRINTERS and all Branches of the Textile Industry

DUDLEY HILL CHEMICAL WORKS BRADFORD

Combined Reports of the Committees on the Dyeing Properties of Direct Cotton, Vat, and Wool Dyes

Price 5/-

Orders should be sent to the

SOCIETY OF DYERS AND COLOURISTS
19 PICCADILLY BRADFORD 1 YORKSHIRE

FORTHCOMING MEETINGS OF THE SOCIETY — *continued from page* xii

Wednesday, 15th February 1956

BRADFORD JUNIOR BRANCH. *Piece Scouring*. B. F. J. Moxon, Esq. (Wool Industries Research Association). Technical College, **Bradford.** 7.15 p.m.

Friday, 17th February 1956

NORTHERN IRELAND SECTION. Dinner Dance. Details later, at Woodbourne House Hotel.

Thursday, 23rd February 1956

MIDLANDS SECTION. *The Finishing of 'Terylene' and 'Terylene'/Wool Blends*. I. E. Haden, Esq., B.Sc., and B. I. Brooke, Esq., B.A.(Oxon.). Gas Theatre, **Nottingham.** 7 p.m.

NORTHERN IRELAND SECTION. *Film Evening*. Thompson's Restaurant, Donegall Place, **Belfast.** (Joint Meeting with the Textile Institute.) 7.30 p.m.

Friday, 2nd March 1956

LONDON SECTION. *Basic Mechanisms of Fading of Dyed Textiles*. H. B. Mann, Esq. (Courtaulds Ltd., Bocking). Royal Society Rooms, Burlington House, Piccadilly, **London, W.1.** 6 p.m.

BRADFORD JUNIOR BRANCH. Annual Dance. Connaught Rooms, **Bradford.**

Friday, 9th March 1956

MIDLANDS SECTION. MIDLANDS SECTION DINNER. Bull's Head Hotel, **Loughborough.**

Tuesday, 13th March 1956

SCOTTISH SECTION. *The Soiling of Synthetic Fibres*. G. G. Taylor, Esq., B.Sc., A.Inst.P. (Clayton Dyestuffs Co. Ltd.). St. Enoch's Hotel, **Glasgow.** 7 p.m.

BRADFORD JUNIOR BRANCH. *Some Aspects of Continuous Vat Dyeing*. Dr. R. J. Rallings (British Cotton Industries Research Association). Technical College, **Bradford.** 7.15 p.m.

Wednesday, 14th March 1956

NORTHERN IRELAND SECTION. *Various Methods of Stripping Vat, Azoic, Direct and Sulphur Dyes*. J. E. Riordan, Esq. (College of Technology, Belfast.) *Effect of pH on the Activity of Bleaching Agents*. J. Boyd, Esq. (College of Technology, Belfast). Thompson's Restaurant, Donegall Place, **Belfast.** 7.30 p.m.

Wednesday, 21st March 1956

MIDLANDS SECTION. *Fast Dyes on Cellulose Triacetate*. J. G. Graham, Esq., B.Sc. (Joint Meeting with British Association of Chemists). Midlands Hotel, **Derby.** 7 p.m.

Thursday, 22nd March 1956

MIDLANDS SECTION. *Dyeing Faults in the Carpet and Hosiery Industries*. G. H. Lister, Esq., B.Sc., Ph.D. (Joint Meeting with the Textile Institute). Carpet Trades Canteen, **Kidderminster.** 7 p.m.

Friday, 23rd March 1956

LONDON SECTION. *Dyeing of Narrow Fabrics—Cellulosic and New Fibres*. Dr. I. M. S. Walls (I.C.I. Ltd.). George Hotel, **Luton.** 7 p.m.

Saturday, 24th March 1956

NORTHERN IRELAND SECTION. Annual General Meeting and Dinner. Details later.

Tuesday, 10th April 1956

SCOTTISH SECTION. *Textile Invention and the Dry Cleaner*. A. J. Crockatt, Esq., M.Sc., F.R.I.C. (Eastmans Dyers and Cleaners Ltd.). St. Enoch's Hotel, **Glasgow.** 7 p.m.

Saturday, 21st April 1956

BRADFORD JUNIOR BRANCH. Annual General Meeting. Technical College, **Bradford.** 10.15 a.m.

Wednesday, 25th April 1956

MIDLANDS SECTION. *A New Approach to High Temperature Dyeing*. S. N. Bradshaw, Esq. Kings Head Hotel, **Loughborough.** Preceded by the Annual General Meeting of the Section. 6.30 p.m.

L J POINTING & SON LTD

Established 1916

HEXHAM

NORTHUMBERLAND

manufacture particularly

SPECIALITY DYESTUFFS

for

OILS, FATS & WAXES
VARNISHES & LACQUERS
LEATHER OF ALL TANNAGES

and

EDIBLE COLOURS
(guaranteed to meet all existing regulations)

ENQUIRIES GIVEN IMMEDIATE AND CAREFUL ATTENTION

Telephones Hexham 942 (3 lines)

Telegrams POINTING HEXHAM

TETRALENE
Detergent and solvent for washing and scouring of textile materials, for use with alkaline or soap solutions

ESTRALENE
Sulphonated Fatty Alcohols in Paste Powder and Liquid

ESTROL
Very efficient wetting and dispersing agent

RETARDOL
For level dyeing of vat colours on rayon etc

FURTHER INFORMATION AND LITERATURE ON REQUEST

Manufacturers of
TEXTILE AUXILIARY CHEMICALS

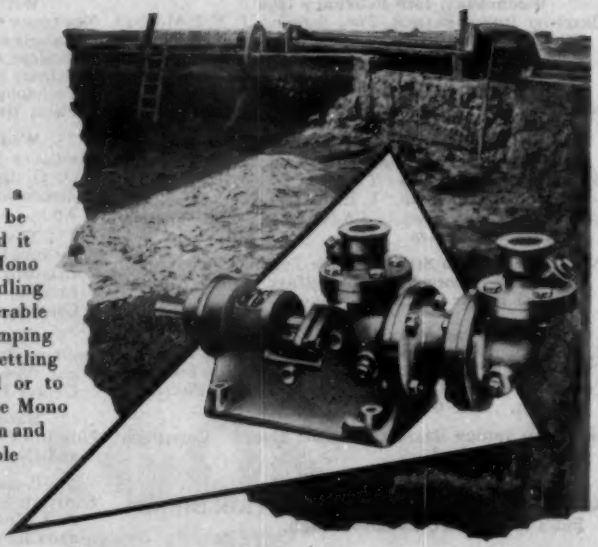
STOCKPORT UNITED CHEMICAL CO LTD

BUXTON ROAD WORKS STOCKPORT

Telephone Great Moor 2980 *Telegrams* TETRALENE STOCKPORT

SLUDGE Disposal

Residue sludge or slurry often creates a major problem of disposal. It may be viscous or carry solids in suspension and it is frequently corrosive or abrasive. The Mono Pump has shown great ability at handling such mixtures and has saved considerable manpower, time and money by pumping sludge effluent direct from pits and settling tanks to transportation for disposal or to plants for by-product recovery. The Mono Pump is self-priming, positive in action and has a high suction power. It is simple and needs the minimum attention.



The MONO pump

MONO PUMPS LIMITED
MONO HOUSE, 1 SEKFORDE STREET, LONDON, E.C.1
Telephone : CLErkenwell 8911
Cables : Monopumps, London *Code :* A.B.C. 7th Edition

MP 242

RONAGEN
COLOURS
for Textile Printing

T SAVILLE WHITTLE LTD
49 Princess Street **MANCHESTER 2**

Aniline & Alizarine Colours
Solvents & Chemicals

We solicit your inquiries for Aniline and Alizarine Colours and Chemicals and shall be pleased to forward samples and prices on application

• **HYDROGEN PEROXIDE**
(CONCENTRATED)
Free from all impurities Prompt deliveries from stock

COLE & WILSON LTD
24 GREENHEAD ROAD HUDDERSFIELD
Telephone Huddersfield 1993 *Telegrams* COLOUR HUDDERSFIELD
WORKS — Common Road Bay Hall Birkby Huddersfield
Telephone Huddersfield 2499

MANOFAST

The MANOFAST PROCESS can now be applied to the following styles and fabrics

DIRECT PRINTING WITH VAT DYESTUFFS ON	DISCHARGE PRINTING WITH VAT DYESTUFFS ON	DISCHARGE PRINTING WITH MORDANT & BASIC DYESTUFFS ON
Cellulose Acetate	Cellulose Acetate	Viscose
Cellulose Acetate & Viscose	Cellulose Acetate & Viscose	Wool
Cellulose Acetate & Wool	Cellulose Acetate & Wool	Real Silk
Cellulose Acetate, Wool, & Viscose	Nylon.	
Wool		
Wool & Cotton		
Nylon		
Terylene.		

- All prints are stable prior to steaming.
- In discharge printing, the trouble of haloing is eliminated.
- There is minimum degradation of the various fibres.
- Excellent colour yields are obtained.

We shall be pleased to discuss technical problems and give practical service on any of the styles and fabrics quoted.

HARDMAN & HOLDEN LTD

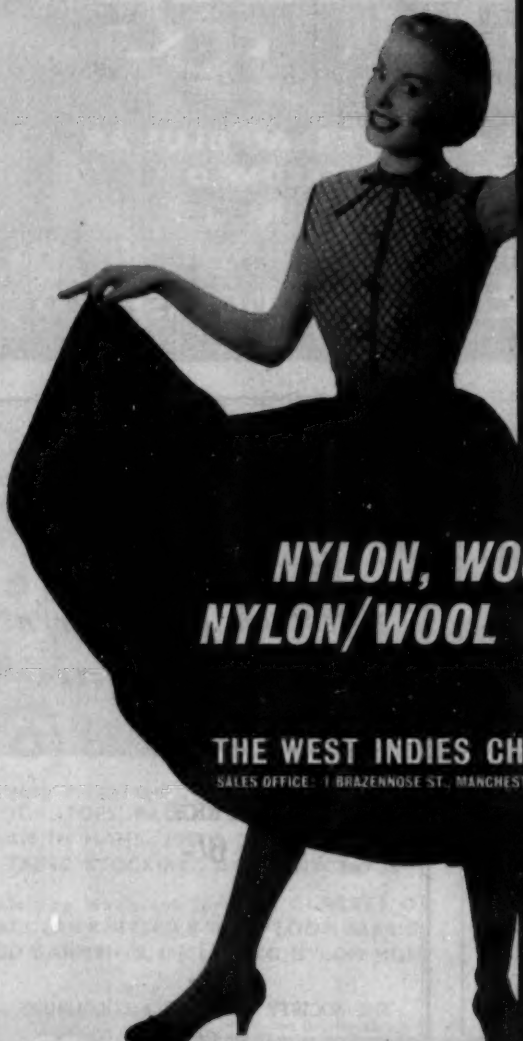
MANOX HOUSE

MILES PLATTING, MANCHESTER 10

Tel. COLlyhurst 1551 (10 lines)

Grams. "Oxide" MANCHESTER

LOGWOOD BLACKS



THE BEST BLACK FOR

NYLON, WOOL AND NYLON/WOOL MIXTURES

THE WEST INDIES CHEMICAL WORKS LTD

SALES OFFICE: 1 BRAZENNOSE ST., MANCHESTER 2. WORKS: HEMATINE, JAMAICA, B.W.I.

TRADE MARK

ALLIGATOR BRAND

C 4440

L·B·H & CO LTD

QUALITY DYES & PRODUCTS

RECENT ADDITIONS

SUPERLAN ASTROL B SUPERLAN BLUE AR

MONOCHROME FAST YELLOW O

SUPERLAN RUBINOL R

FOR SAMPLES AND QUOTATIONS APPLY—

L B HOLLIDAY & CO LTD HUDDERSFIELD

BROWN & FORTH

LIMITED

FOUNDED IN 1890

Sole Distributors in the U K & Eire

of the

SEVRON

DYESTUFFS

specially manufactured by

E I du Pont de Nemours & Co

for their

ORLON*

ACRYLIC FIBRE

* du Pont trade mark

83-117 EUSTON ROAD LONDON NW 1

EUS 5101-5

81 CORNBROOK STREET MANCHESTER 16

MOS 1347-8

AND AT NEW YORK

GEOMETRIC GREY SCALE

for Assessing the Effect on the Pattern in Fastness Testing

PRICE

6/-

from

THE SOCIETY OF DYERS & COLOURISTS

19 PICCADILLY

BRADFORD 1 YORKSHIRE ENGLAND

PEGG

High temperature TOP DYEING MACHINE

Built in a range of capacities from 1-64 Tops

Dyeing Machines of all types for LOOSE STOCK, TOPS, PACKAGES, BEAMS, CAKES, YARN IN HANK, PIECE-GOODS, RIBBONS, TAPES, STOCKINGS AND KNITGOODS

Finishing Machines for ALL CLASSES OF CIRCULAR KNITTED & WARP LOOM FABRICS AND GARMENTS, INCLUDING NYLON HOSE

STEVERLYNCK PROCESS

Built within the Bentley Group

SP 25

PEGG

SAMUEL PEGG & SON LTD.

BARKBY ROAD · LEICESTER · ENGLAND

TELEPHONE 67884 5 TELEGRAMS THRUSTOR LEICESTER

Notice to Authors of Papers

The MSS. of all papers communicated to or read before the Society become the property of the Society. They should be addressed to the Editor, Society of Dyers and Colourists, 19 Piccadilly, Bradford 1. Authors must not allow their papers to be published elsewhere before they have appeared in the Society's *Journal*. Should prior publication take place without the sanction of the Publications Committee, the paper will be printed only as an abstract or summary.

Manuscripts submitted for publication in the *Journal* should be typewritten (double spacing) on good-quality paper, using one side of the paper only and leaving a margin at least 1 in. wide on the left-hand side. The time taken in refereeing papers (both lectures and communications) will be reduced to a minimum when authors submit two copies of the typescript.

In view of the high costs of publication, it is essential that authors should be as concise as possible. When experimental procedure has already been published, a literature reference to the paper containing the details is sufficient, whilst well known experimental methods should be described very briefly.

Introductory paragraphs describing the aims of the investigation and the method of attack are desirable, and should be followed by the experimental results and their discussion. There should be a brief summary for insertion at the beginning of the paper. References to the literature should be numbered consecutively, using superscript numbers without brackets immediately following the text words or author's name to which they refer.

The list of references should be given at the end of the manuscript and the abbreviations used should be, as far as possible, those given in the "List of Periodicals Abstracted" included at the end of the index to the preceding year's *Journal*. Reference numbers in this list should be neither enclosed in brackets nor followed by full-stops. As far as possible throughout the manuscript the abbreviations listed in the Jan. 1950 issue (p. 54) should be used. Tables should be numbered consecutively in Roman numerals and Figures in Arabic numerals.

The number of figures and graphs should be kept as low as possible, and data should be presented in the form of either tables or graphs, not both. Drawings should be carefully prepared, preferably in Indian ink, on plain white drawing paper or, preferably, Bristol board. In graphs, the frame and actual curves should be ruled and inked more heavily than any co-ordinate lines, and the latter should not be close together as in ordinary graph paper. Experimental points should always be given, and where several graphs appear in a single Figure clear means of differentiation must be adopted. All numbers and legends are set up in type by the printer, and authors should therefore indicate them lightly in pencil.

Twenty-five free copies of a reprint are supplied to the author of an original paper published in the *Journal*, or fifty free copies are supplied when there are two or more authors, and a further number may be purchased from the Society at the rates given below.

REPRINTS OF LECTURES AND COMMUNICATIONS

Reprints of all lectures and communications are available after publication to members and non-members of the Society. The charges (postage included) are as follows— Single copies 2s. 6d. each; per dozen copies up to and including 8 pages, 12s. 6d., and for papers occupying more than 8 pages of the *Journal*, 17s. Orders should be addressed to "The Society of Dyers and Colourists, 19 Piccadilly, Bradford 1". They can be accepted only if accompanied by remittance and if received immediately after publication of the paper.

INDEX TO ADVERTISERS

Page	
xxxii	Albright & Wilson Ltd
xxiv	Alcock (Peroxide) Ltd
xxv	Bayer Leverkusen Germany
xxvi	B.I.P Chemicals Ltd
xxi	Brotherton & Co Ltd
xxxviii	Brown & Forth Ltd
xix	Ciba Ltd
xvii	Clayton Dyestuffs Co Ltd
xxxv	Cole & Wilson Ltd
ix	Colne Vale Dye & Chemical Co Ltd
x	Chas. Forth & Son Ltd
i	Gardinol Chemical Co Ltd
xxxvi	Hardman & Holden Ltd
xxxviii	L B Holliday & Co Ltd
viii	Howards of Ilford Ltd
xiv	Imperial Chemical Industries Ltd
xx	Imperial Chemical Industries Ltd
vii	Johnsons of Hendon Ltd
iii	Laporte Chemicals Ltd
vi	John W Leitch & Co Ltd
xxviii	Longclose Engineering Co Ltd
ii	Mather & Platt Ltd
xxxiv	Mono Pumps Ltd
vii	Newell (Chemicals) Ltd
xxxix	Samuel Pegg & Son Ltd
xxxiv	L J Pointing & Son Ltd
xviii	James Robinson & Co Ltd
xiii	Sandoz Products Ltd
xv	Sandoz Products Ltd
iv	Saunders Valve Co Ltd
xxxv	T Saville Whittle Ltd
xi	Shell Chemicals Ltd
iii	Shirley Developments Ltd
xvi	F Smith & Co (Whitworth) Ltd
ix	Standard Chemical Co
i	Staveley Iron & Chemical Co Ltd
xxxiv	Stockport United Chemical Co Ltd
xxii	Joseph Stubbs Ltd
xxvii	Tennants Textile Colours Ltd
xxix	W P Thompson & Co
xxxvii	West Indies Chemical Works Ltd
v	Whiffen & Sons Ltd
xxxiii	J B Wilkinson (Chemicals) Ltd
xxiii	Yorkshire Dyeware & Chemical Co Ltd

PUBLICATIONS OF THE SOCIETY OF DYERS AND COLOURISTS

(All publications are sent post free)

JOURNAL OF THE SOCIETY OF DYERS AND COLOURISTS
(published monthly) Price to Non-members per annum £4

THE THEORY AND PRACTICE OF WOOL DYEING (Second Edition)
By C L BIRD *Price* 15*s*

IDENTIFICATION OF DYES ON TEXTILE FIBRES AND DETECTION OF METALS IN FIBROUS MATERIALS, DYES, AND ORGANIC PIGMENTS (Second Edition)
By ELLIS CLAYTON *Price* 10*s* 6*d*

REPORTS OF THE COMMITTEES ON THE DYEING PROPERTIES OF DIRECT COTTON, VAT, AND WOOL DYES
Price 5*s*

TEXTILE PRINTING
Proceedings of a Symposium held at St. Annes-on-Sea in September 1953
Price £1 10*s*

THE TINCTORIAL ARTS TO-DAY
Proceedings of a Conference held at Harrogate in September 1951
Price 15*s* Members £1 5*s* Non-members

PHOTOCHEMISTRY IN RELATION TO TEXTILES
Proceedings of a Symposium held at Harrogate in September 1949
Price £1 Members £1 10*s* Non-members

RECENT ADVANCES IN THE THEORY AND PRACTICE OF DYEING
Unbound Volume of the Proceedings of a Symposium held at Blackpool in September 1947
Price 15*s*

REVIEW OF TEXTILE PROGRESS
Volume I 1949 £1 Volume II 1950 £1 5*s*
Volume III 1951 Volume IV 1952 Volume V 1955 £1 15*s*
(£1 8*s* to S.D.C & T.I Members)

A S D C EXAMINATION 1954
QUESTION PAPERS AND REPORT OF THE DIPLOMAS EXECUTIVE SUB-COMMITTEE
Reprinted from the Journal of the Society (February 1955)
Price 1*s*

THE SOCIETY OF DYERS AND COLOURISTS
19 PICCADILLY BRADFORD 1 YORKSHIRE ENGLAND

PUBLISHED FOR THE SOCIETY AND PRINTED BY GEORGE AND PICKERSGILL LTD LEEDS
ENTERED AT THE NEW YORK POST OFFICE AS SECOND CLASS MATTER